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# Hydrocarbon Fuels for Advanced Systems

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Shell Development Company  
A Division of Shell Oil Company  
Emeryville, California

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HYDROCARBON FUELS FOR  
ADVANCED SYSTEMS

G. H. Ackerman, L. E. Faith, C. K. Heck, H. T. Henderson,  
A. W. Ritchie, and L. B. Ryland

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## FOREWORD

The work described in this technical report was authorized under J.S. Air Force Contract No. F33615-70-C-1038, Project 3048. The program was administered by Mr. H. R. Lander and Lt. J. C. Ford, Project Engineers, Air Force Aero Propulsion Laboratory/APPF-1, Wright-Patterson Air Force Base, Ohio 45433.

This program is being conducted at Shell Development Company, Emeryville, California 94608 under the general supervision of K. D. Detling, Manager, Government Research Department. This report is the first annual technical report under Contract No. F33615-70-C-1038. It was submitted on September 17, 1970. The report describes the results of work performed during the period 1 September 1969 to 31 August 1970. This investigation represents a continuation of a previous study, "Vaporizing and Endothermic Fuels for Advanced Engine Application" under Contract No. AF 33(615)-3789, the results of which were published in Technical Report AFAPL-TR-67-114, Part I, October 1967, Part II, September 1968; Part III, February 1970.

Acknowledgment is made to A. C. Nixon for many valuable contributions to the program as principal investigator and project supervisor until his retirement on 1 June 1970.

This technical report has been reviewed and approved.

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## ABSTRACT

The general objective of this investigation is the development of new fuels and new fuel systems which will provide the cooling and propulsion requirements of advanced air-breathing engines. In previous studies on the utilization of endothermic reactions of hydrocarbon fuels, the catalytic dehydrogenation of naphthenes showed the most promise for practical applications. In continuing these studies, a large number of catalysts were prepared and tested for activity and stability for the dehydrogenation of methylcyclohexane and decalin. The stability of supported platinum catalysts for these reactions was affected by the physical properties and chemical composition of the support and by the metal content and metal composition of the catalyst. Bicyclo(2,2,2)octane was dehydrogenated to bicyclooctene in low yield with a supported platinum catalyst. Additives were found which enhanced the rate of thermal cracking of paraffins. Improved wall catalysts were formulated for the dehydrogenation of naphthenes and a mathematical model was developed for a catalytic wall reactor. Various fuels were evaluated for thermal stability in both our standard ASTM Coker and a special Alcor JFTOT unit designed for operation up to 1000°F and 1000 psi. The high temperature thermal stability of fuels was improved by means of additives. The deposit profiles on coker tubes were reproducibly determined with a recently developed beta-ray backscatter instrument which is capable of measuring deposit thicknesses up to 2500 Å. The physical properties of JP-7 jet fuel were recalculated using improved methods. The new values compare favorably with data obtained from heat transfer experiments. The supersonic combustion of decalin, tetralin and naphthalene was investigated using the shock tube. A literature survey was made of articles and patents of interest to this and related programs.

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## HYDROCARBON FUELS FOR ADVANCED SYSTEMS

INTRODUCTION

As vehicles propelled by airbreathing engines are being designed to fly at greater speeds, thermal problems multiply and cooling needs increase. The most critical regions which require cooling are leading edges and engines. Although thermal effects can be somewhat accommodated by improved materials and passive cooling, sustained hypersonic flight in the atmosphere requires a substantial heat sink. Mechanical refrigeration or a noncombustible coolant can be used for cooling, but the fuel remains as the most efficient source of heat sink.

The speed limit on vehicles in the range above Mach 4 will depend on the cooling capacity of the fuel. Fuels such as hydrogen, methane, and ammonia can furnish a heat sink only through sensible heating and vaporization, whereas hydrocarbon fuels can provide additional cooling through endothermic reactions. Hydrocarbons can undergo both thermal (noncatalytic) and catalytic endothermic reactions. Theoretically the total heat sink of hydrocarbon fuels range from 50 to 112% of the cooling capacity of hydrogen, based on heat sinks normalized by the heat of combustion. Laboratory proven capability has achieved 85% for catalytic reaction accompanied by sensible heating and vaporization, whereas thermal reaction, such as cracking, with heating and vaporization has been limited to 55%.

Catalytic reactions of interest include dehydrogenation, dehydrocyclization, and depolymerization. The most promising type that has been studied is the catalytic dehydrogenation of naphthenes. A typical example of this reaction is the dehydrogenation of methylcyclohexane over a platinum on alumina catalyst. Other promising reactions are the catalytic dehydrogenation of dicyclohexyl and decahydronaphthalene.

Until a few years ago the use of hydrocarbon fuels in advanced engines at speeds greater than Mach 3 was no more than a concept. Studies under contracts AF33(657)-11096 and AF33(615)-3789 investigated the possibility of using hydrocarbon fuels under the severe conditions of hypersonic flight. These programs were directed toward evaluating the capability of these fuels in advanced engines and developing the information necessary for the design of systems for the promising fuels.

The current contract is a continuation of these past programs with emphasis on extending the knowledge and improving the capability and performance of known hydrocarbon fuels. In addition, investigations of new candidate fuels and new techniques for utilizing hydrocarbon fuels are continuing.

Areas of investigation in the current program are the study of endothermic reactions and the determination of properties and characteristics of hydrocarbon fuels. The catalytic and thermal reactions of different fuels are being investigated and the kinetics of reactions with significant heat sinks are being measured and analyzed. Catalysts are being studied with the objective of optimizing the composition and geometry so as to improve catalyst activity, stability, and performance. Properties and characteristics of fuels are being determined experimentally and theoretically. Gas properties can be predicted reliably, whereas liquid properties must be measured. Thermal stability and combustion characteristics of fuels are being determined by experimental work.

## SUMMARY

As hypersonic aircraft and missiles are developed with increasing speed, the need for engine cooling multiplies. The fuel used in an engine is a convenient coolant for this purpose, absorbing heat as sensible heat and latent heat of vaporization. Certain hydrocarbon fuels can furnish additional heat sink in the form of endothermic reactions. Possible endothermic reactions include thermal reactions such as cracking, and catalytic reactions such as dehydrogenation, dehydrocyclization, and depolymerization. Of these, the catalytic dehydrogenation of naphthenes to aromatics is currently the most promising type of reaction. Reactions of this type are very selective and proceed rapidly to achieve high conversion. The total heat sinks for these reactions compare favorably with heat sinks for other fuels.

Over forty-five catalysts were evaluated in bench-scale studies for stability in the dehydrogenation of naphthenes without added hydrogen. Almost all of the catalysts contained platinum either alone or with other metals as bimetallic or trimetallic mixtures; these were mounted on eighteen different supports. In short tests with decalin (30 minutes) at 10 atm pressure, six catalysts showed moderate stability at 1200°F and good stability at lower temperatures. Four of these catalysts contained platinum on four different supports; the other two were bimetallic and trimetallic mixtures. In less severe tests with MCH nine of the catalysts showed moderate stability at high space velocity (LHSV = 100). Based on the tests with decalin it appeared that catalyst stability was affected by catalyst support composition, physical properties of the support, and the catalyst metal content and composition.

Exploratory studies with bicyclo(2,2,2)octane in a pulse reactor showed that this naphthene could be catalytically dehydrogenated to bicyclooctene. Yields were low (ca 26%) and the reaction was quite sensitive to catalyst properties and reaction conditions. With one catalyst bicyclooctene was formed using He carrier gas but not with H<sub>2</sub> carrier; with two other catalysts containing platinum, bicyclooctene was formed with one catalyst but not with the other.

Twenty different additives were tested in the pulse reactor as free radical initiators for enhancing the rate of the thermal cracking reaction. Of the additives tested six gave substantial increases in the rate of cracking of n-dodecane at 1100°F. The most effective additive caused an increase in conversion of about 75%.

Addition of 50% propane to methylcyclohexane enhanced the reactivity of MCH at and below 932°F but reduced reactivity at higher temperatures.

Development of granular and coating catalysts and screening of catalysts for dehydrogenation activity have continued. Many additional granular catalysts have been prepared which consist of one or more metals on various supports or mixtures of several unsupported metals. Development of catalytic coatings for metal surfaces have continued in order to improve ease of application, mechanical properties, adherence to metal surfaces, and catalytic activity. Candidate coatings in platinized granular form have been evaluated in tests in the micro catalyst test reactor.



The need for obtaining intrinsic reaction kinetic data in the absence of heat and mass transfer effects has been discussed. Packed bed reactors have a number of drawbacks and some kind of recycle reactor must be used. A catalytic continuous stirred tank reactor is appropriate and one has been designed to measure the kinetics of the dehydrogenation of methylcyclohexane and decalin.

A mathematical model of the catalytic wall reactor configuration has been developed. The model is one-dimensional in fluid flow, but accounts for radial heat fluxes. Differential equations describing temperature, pressure and conversion are integrated numerically along the reactor. The experimental data on methylcyclohexane dehydrogenation obtained in the Fuel System Simulation Test Rig (FSSTR) are discussed and compared with the model. After slight modification of the kinetic parameters, the model agreed well with the experiments. Approximations and possible modifications of the model are also considered.

Test runs were made on the dehydrogenation of decalin with three different catalysts in the FSSTR. Shell 10860-146 catalyst was far superior in activity and stability to Shell 10280-113 and UOP-R8 catalysts at conversions up to 80% and outlet fuel temperatures up to 1050°F. The two latter catalysts were comparable in initial activity, but Shell 113 catalyst was much less stable than UOP-R8 catalyst at higher temperatures.

A problem in the determination of fuel thermal stability by a heat transfer device such as the ASTM Coker is the uncertainty in evaluating both the amount and the distribution of the deposits on the tube at the conclusion of the test. While it is usually possible to rate deposits by appearance on highly polished aluminum tubes, many other metals such as stainless steels and nickel change color when heated, even in inert environments, and this complicates the visual rating. To solve this problem we have built a beta-ray backscatter instrument which is capable of evaluating deposit thicknesses up to 2500 Å. We have demonstrated that the deposit profiles on coker tubes can be reproducibly determined. The calibration of this instrument on aluminum tubes has been accomplished, and similar calibrations on stainless steels and other metals are in progress. We have measured deposits from several sources, and in many cases the geographical distribution of deposits measured by this instrument is the same as determined by the optical methods of evaluation. However, the relative values vary greatly, and in some cases even the geographical distributions are different. In fact, it appears at this time that the beta-ray backscatter instrument is capable of giving a truer picture of deposit distribution than the optical method.

One recent addition to the battery of equipment being used for evaluation of fuel thermal stability is a special Alcor JFTOT unit, designed for operation up to 1000°F and 1000 psi. Although the equipment has not been operated as yet up to its maximum capability, considerable data have been obtained with it under less severe conditions with excellent results. Good agreement has been found between data obtained with this equipment and those obtained with our standard ASTM Coker. However, some difficulty has been encountered at high temperatures due to a tendency of the thin aluminum tubes to buckle. Attempts to correct this by the use of TEFLON® O-rings and a conducting grease at the bus-bar clamps were not successful.

About 50 compounds have been screened on the SD/M-7 Coker for their possible use as fuel additives in improving high temperature thermal stability. Six of these have been found effective in the liquid temperature range of 675 to 700°F, and the best of them increases the thermal stability breakpoint by 125°F liquid or 160°F metal temperature.

A storage stability experiment on high purity MCH, which was initiated about 4-1/2 years ago, has been terminated with the removal of the samples from the hot room and their examination. The samples were stored in one-gallon epoxy-lined pails with a 2:1 gas/liquid ratio at 130°F, the gas compositions ranging from pure oxygen to pure nitrogen. The remarkable result was that all the samples appeared equivalent to their original condition; no deterioration had occurred that could be ascertained by color change, gum determinations, or thermal stability testing with the SD/M-7 Coker. Some tests have been made for the selection of the most favorable antioxidant for SHELLDYNE-H® hydrocarbon fuel. Samples were tested with various commercial inhibitors under accelerated conditions, 200°F in the presence of excess oxygen for 18 and 30 hours. Although the results were not clear because of difficulties with gum determinations, the most effective antioxidant for SHELLDYNE-H® fuel appears to be the amine inhibitor, di-sec-butyl p-phenylene diamine.

A new batch of MCH, synthesized by toluene hydrogenation, has been tested in the SD/M-7 Coker, and is now being filtered to meet thermal stability specifications.

Revised physical properties for JP-7 jet fuel were calculated by improved predictive methods. Properties were estimated for saturated gas and liquid at subcritical temperatures and for the gas at supercritical temperatures and different pressures. These revised values are considered to be better than previous properties, especially for the liquid heat capacity and thermal conductivity. The revised properties agree quite well with properties of similar mixtures, and compare favorably with data from heat transfer experiments.

Physical properties were estimated for binary mixtures of methylcyclohexane (MCH) and a low molecular weight hydrocarbon (ethane, propane and n-butane). These were prepared for use in future studies on fuel cooling of advanced engines. Mixture properties were determined from known properties of the individual constituents. Melting and freezing points of MCH-propane mixtures were measured and used to calculate melting point depressions for the desired mixtures.

The surface tension of SHELLDYNE-H® hydrocarbon fuel was measured at three temperatures. These results were correlated, and the correlating equation was used to estimate surface tension at higher temperatures.

The supersonic combustion of decalin, tetralin and naphthalene were investigated using the shock tube. Methods of accounting for attenuation were compared, and the best one was found to be the use of a constant value (0.001) for the attenuation coefficient. The ignition delay time correlation was modified by adding the effect of fuel concentration. The data obtained on decalin and tetralin and much of the data on ignition delay times obtained earlier were correlated using the new equation. A statistical analysis showed that the correlations could not be analyzed by standard techniques. The method of fitting the parameters in the equation to

the experimental data was also discussed. In general, it is seen that oxygen has the effect of reducing the ignition delay times, while increases in fuel concentration tend to increase it. The rate of combustion of tetralin was also measured. From limited data it seems that temperature, oxygen, and fuel all tend to increase the rate of combustion. As has been observed before, the activation energy is fairly low.

Pertinent articles and patents that appeared in the literature during the past year were compiled into a bibliography. This survey covered the following subjects: Advanced Fuels, Physical and Chemical Properties of Fuels, Thermal Stability of Fuels, Fuel Contaminants and Additives, Catalysts and Catalytic Reactions, Heat Transfer and Fluid Mechanics, Combustion, and Advanced Engine Development.

## LABORATORY STUDIES OF CATALYTIC AND THERMAL REACTIONS

The bench-scale studies of candidate endothermic fuels and their catalyst systems that were initiated under the previous contract are being continued. Tests were conducted in apparatus developed under the previous contract.<sup>1)2)3)</sup>

Extending the work on the stability of catalysts for the dehydrogenation of naphthenes, over forty-five catalysts were evaluated using the dehydrogenation of decalin and of methylcyclohexane as the test reactions in our bench-scale system. The dehydrogenation of bicyclooctane was studied in a pulse reactor as part of our exploratory search for additional endothermic fuels. A number of additional additives were tested as free radical initiators for the thermal cracking of n-dodecane.

### Bench-Scale Catalyst Stability Studies

Work under the previous contract showed that there were considerable differences in the stabilities of supported platinum catalysts for the dehydrogenation of naphthene reactions.<sup>2)3)</sup> Further, preliminary studies with a few commercial and laboratory catalysts showed that stability was strongly affected by the catalyst pore structure.<sup>3)</sup> As the most efficient catalyst for naphthene dehydrogenation appears to be platinum, it was of interest to expand the study of the stability of this catalyst system. The purpose of the work was to study the variables that influenced stability, with the subsequent aim of optimizing the catalyst composition.

Under the present contract about forty-five catalysts have been evaluated for stability using the dehydrogenations of decalin (DHN) and methylcyclohexane (MCH) as test reactions. Three of these catalysts contained no platinum. The remainder consisted of fourteen different elements combined with platinum as bimetallic or trimetallic mixtures or compounds and mounted on twelve different supports. The variables under study were composition of catalyst support, physical properties of the support, metal composition, metal content, and metal dispersion. A detailed description of the preparation of these catalysts is given in a later section.

The tests were done in our bench-scale laboratory reactor system which was a tubular flow reactor equipped with conventional devices for measuring feed flow rates and for collecting liquid and gas products. The reactor was a stainless steel tube (No. 347, 1/2-in. IPS) 32-in. long and 5/8-in. ID., which was heated by an electric furnace. The catalyst was contained in the annular space between the thermowell and the reactor wall. In order to supply heat rapidly to the catalyst bed, the annular distance between the thermowell and the reactor wall was about 1/16 in., which was about one-pellet diameter. The catalyst bed was about 4-1/2-in. long and had a volume of 7 ml. Prior to carrying out the experiments, the catalysts were reduced in situ with hydrogen for 30 minutes at 572°F (300°C) and then for one hour at the reaction temperature. The complete apparatus was described in detail in a previous report.<sup>3)</sup>

The reactor wall temperature was measured by a thermocouple pressed against the outside reactor wall by the furnace block and located about 1 in. below the top of the catalyst bed. The catalyst bed temperatures were measured by thermocouples contained in the thermowell. The thermocouples were 1 in. apart and the top thermocouple was about 1/2 in. below the top of the catalyst bed (the flow was downward through the catalyst bed). The "effective" catalyst temperature was between the reactor wall temperature and the catalyst bed temperature.

During reaction the catalyst bed temperature (thermocouple measurements) was considerably lower than the furnace block temperature due to the endothermic heat of reaction. As the catalyst deactivated the catalyst bed temperature increased and the magnitude of the temperature increase was taken as a measure of catalyst deactivation. Another quantitative indication of catalyst deactivation was the movement of the "cold spot" down the catalyst bed.

Product analyses were done by GLC from which conversions and selectivities were calculated.

#### Dehydrogenation of Decalin

The catalysts were tested at 10 atm pressure and a liquid hourly space velocity (LHSV, volume of feed per volume of catalyst per hour) of 100. Each catalyst was tested initially at 842°F, and then at successively higher temperature (in 90°F increments) through 1202°F or until the catalyst became inactive. The test period was 30 minutes at each temperature. The feed (F-113 DHN) had the following composition:

25.0% trans-DHN  
74.6% cis-DHN  
0.4% tetralin (THN)

The catalysts were tested in two groups, one of which contained catalysts prepared under the previous contract for another purpose, and the other which contained catalysts prepared under this contract. (The preparative methods are described in a later section.)

The principal reaction products were tetralin (THN) and naphthalene (N). With fresh catalyst high selectivities (90-100%) to THN and N were observed. When the catalysts became highly deactivated side reactions occurred that gave products which emerged from the GLC before and after trans-DHN, after cis-DHN, and after N. These products were not identified further, although those emerging before trans-DHN were assumed to be cracked material (i.e., lighter than DHN). Some cis to trans-DHN isomerization was observed at the lower temperatures (842° and 932°F) with all of the catalysts. The complete data, including product analyses, are shown in Table 55 in the Appendix.

In the first series of runs, fifteen catalysts were tested that had been prepared under the previous contract. These consisted of pure platinum, platinum combined with twelve other metals as bimetallic or trimetallic mixtures, and three different non-platinum bimetallic mixtures. The metals were mounted on four different supports. The pertinent data are summarized in Table 1, which also includes data for a few catalysts tested previously. Figures 1 and 2 show activity (i.e., conversion) and deactivation (i.e., increase in catalyst temperature,  $\Delta T_{\text{max}}$  °F) as functions of block temperature.

The most stable catalyst in this series was Shell 157B (10280-157B). This catalyst was a bimetallic platinum mounted on a granular Type 1 support. The stability (Figure 2) was about that of Shell 46 (granular Type 1 support) and 114C (granular Type 6 support) but the activity was lower than that of the latter two catalysts (Figure 1). Another catalyst with good to moderate stability was Shell 15A (10280-15A), a trimetallic platinum mounted on a spherical Type 1 support. In fact this catalyst was the most stable of all of the catalysts containing this support that have been tested thus far. UOP-R16E, which is the UOP "high stability" platforming catalyst, did not deactivate at 1112°F, but did show a catalyst temperature increase of 104°F compared to only 36°F for Shell 15A. However, the activity of this latter catalyst was lower than that of UOP-R16E (Figure 1). Thus Shell 15A was more stable but less active than UOP-R16E.

The remainder of the catalysts in this series showed poor stability at the higher temperatures (Table 1, Figure 2).

In a second series of runs eighteen different catalysts prepared under the present contract were tested. These catalysts were pure platinum or platinum plus another element that were mounted on ten different supports. The preparation of these catalysts is described in a later section. The pertinent test data are shown in Table 2.

Of the catalysts tested in this series, Shell 135B and 142B were the most stable. Thus moderate stabilities with increases in catalyst bed temperatures of 47° and 56°F, respectively, were observed at 1202°F (block) and good stabilities at lower temperatures. The other catalysts showed poor stabilities at 1202°F ranging from complete deactivation to temperature increases of 131 to 227°F (Table 2). Figure 3 shows stability as a function of block temperature for a number of these catalysts. The performance of Shell 135B and 142B was comparable to that of Shell 46 and Shell 114C (Table 2) and these four catalysts together with 157B are the most stable of those tested thus far. Actually Shell 135B and Shell 46 are nearly identical catalysts. They contained the same support and same Pt content, but the method of mounting the platinum on the support was different. One of the catalysts contained chloride and the other was chloride-free. Within the limits of our tests the performance of these catalysts was the same.

Shell 146 (10860-146) was a large batch of catalyst (500g) prepared in same manner as was Shell 46 and is intended for use in the FSSTR. In our tests the performance of this catalyst was similar to that observed with Shell 46, possibly a little more active and a little more stable than the latter, based on the test data at 1112°F (Table 2). Unfortunately it was not possible to complete the test at 1202°F with Shell 146 due to plugging of the liquid receiver with naphthalene. This suggests that Shell 146 was a little more active (i.e., gave higher conversion to naphthalene) than Shell 46 as the receiver did not plug in tests with this latter catalyst.

Activities (as measured by conversions) were different for the various catalysts but in general the more active catalysts were the more stable.

Tables 1 and 2 and  
Figures 1 through 3 follow

Table 1. DEHYDROGENATION OF DECALIN OVER VARIOUS CATALYSTS: FIRST SERIES.

Catalyst Stability Tests

Pressure: 10 atm  
LHSV: 100  
Reaction Time: 30 minutes  
Feed: F-113 DHN  
Feed Composition: 25.0% trans DHN  
74.6% cis DHN  
0.4% THN  
Catalyst Volume: 7 ml

Catalyst Number	DHN Conversion, %w at					Increase in Catalyst Temperature, $\Delta T_{max}$ , °F at				
	842°F	932°F	1022°F	1112°F	1202°F	842°F	932°F	1022°F	1112°F	1202°F
10280-46 <sup>a)</sup>	41.9	50.0	59.2	67.9	81.2	13	14	16	24	65
10280-157B	29.8	37.9	47.2	56.3	67.9	16	22	27	31	58
10280-119C	37.5	47.0	56.3	67.0	77.1	13	14	16	23	171
10280-124C	25.3	34.0	43.2	53.4	46.5	20	29	27	40	234
10280-15A	30.6	38.3	48.2	59.3	66.9	16	23	25	36	107
9874-193A	29.3	36.4	42.9	43.2	-	17	45	154	272 <sup>b)</sup>	-
10280-13	30.3	37.9	41.3	24.0	-	14	38	238	281 <sup>b)</sup>	-
10280-50A	9.6	5.7	10.2	-	-	126	-	-	-	-
9874-194A	29.8	37.9	40.5	-	-	18	43	256 <sup>b)</sup>	-	-
9874-192A	29.0	35.2	30.4	-	-	22	157	252 <sup>b)</sup>	-	-
9874-186A	29.7	36.3	37.0	-	-	20	74	261 <sup>b)</sup>	-	-
9874-189A	27.3	33.4	23.2	-	-	16	92	248 <sup>b)</sup>	-	-
9874-187A	27.8	32.0	26.6	-	-	20	101	257 <sup>b)</sup>	-	-
9874-194B	21.5	25.8	25.6	-	-	128	315 <sup>b)</sup>	-	-	-
9874-199B	19.2	22.8	20.7	-	-	140	191	194 <sup>b)</sup>	-	-
UOP-R16 <sup>a)</sup>	31.1	42.1	52.9	61.7	-	5	11	22	104	-
UOP-R8 <sup>a)</sup>	36.2	43.9	47.7	-	-	14	43	160	-	-
10850-114C <sup>a)</sup>	41.8	50.5	62.0	71.7	83.0	5	2	4	16	67
10280-77A	33.1	47.2	57.0	64.3	72.8	13	14	22	70	229 <sup>b)</sup>
9874-119B	39.3	47.1	55.8	62.6	57.4	14	22	45	133	339 <sup>b)</sup>

a) Previously tested.

b) Catalyst completely deactivated.

Table 2. DEHYDROGENATION OF DECALIN OVER VARIOUS CATALYSTS: SECOND SERIES

Catalyst Stability Tests

Pressure: 10 atm  
LHSV: 100  
Reaction Time: 30 minutes  
Temperatures: Block

Feed: F-113  
Feed Composition: 25.0% trans DHN  
74.6% cis DHN  
0.4% Tetralin

Catalyst Number	Support Pore Diameter, Å	% DHN Conversion at, °F					ΔT, °F of Catalyst Bed at, °F				
		842	932	1022	1112	1202	842	932	1022	1112	1202
10280-44 <sup>d)</sup>	106	33.5	42.7	51.4	-	-	7	20	43	-	-
10860-132A	176	33.2	39.5	33.7	-	-	7	58	248	-	-
-134A	176	26.2	31.1	19.4	-	-	27	94	182 <sup>a)</sup>	-	-
-134B	176	27.2	34.5	33.4	-	-	11	56	241	-	-
10860-135A	40	34.8	42.6	52.2	64.9	72.0	16	9	27	31	131 <sup>a)</sup>
-132C	40	35.5	43.7	53.5	61.7	68.8	14	20	25	40	- <sup>a)</sup>
10280-114B	-	33.1	44.9	53.2	60.6	64.1	14	16	43	108	-
10860-132B	176	33.5	44.0	55.5	66.6	69.2	5	9	32	95	227 <sup>b)</sup>
10860-138B	106	37.2	45.0	55.2	65.9	32.2	23	20	31	97	- <sup>a)</sup>
-135D	50	37.2	48.0	55.2	63.7	73.2	11	16	7	23	142
-134D	65	36.1	46.7	51.1	62.6	71.2	7	4	11	23	146
-142B	28	41.4	50.7	56.3	71.2	77.6	5	4	7	36	56
-137B	70	29.7	39.6	48.5	56.0	-	13	23	41	67	-
10280-46	40	41.9	50.0	59.2	67.9	81.2	13	14	16	24	65
10860-139B	-	30.3	40.0	44.7	59.5	52.2	18	23	56	139	- <sup>a)</sup>
-143B	-	28.9	33.6	20.4	-	-	47	160	- <sup>a)</sup>	-	- <sup>c)</sup>
10860-146	40	43.7	53.8	62.2	71.2	84.1 <sup>c)</sup>	3	5	13	18	- <sup>c)</sup>
-147	-	30.0	36.7	43.2	44.2	-	13	22	40	135	-
-135B	40	42.4	53.7	62.6	70.0	77.9	6	5	9	12	47
-163A	-	39.3	44.7	54.3	63.7	45.5	7	11	37	48	290

a) Catalyst completely deactivated after 10-15 minutes at this temperature.

b) Catalyst completely deactivated at end of run.

c) Reaction time 10 minutes.

d) Standard Catalyst



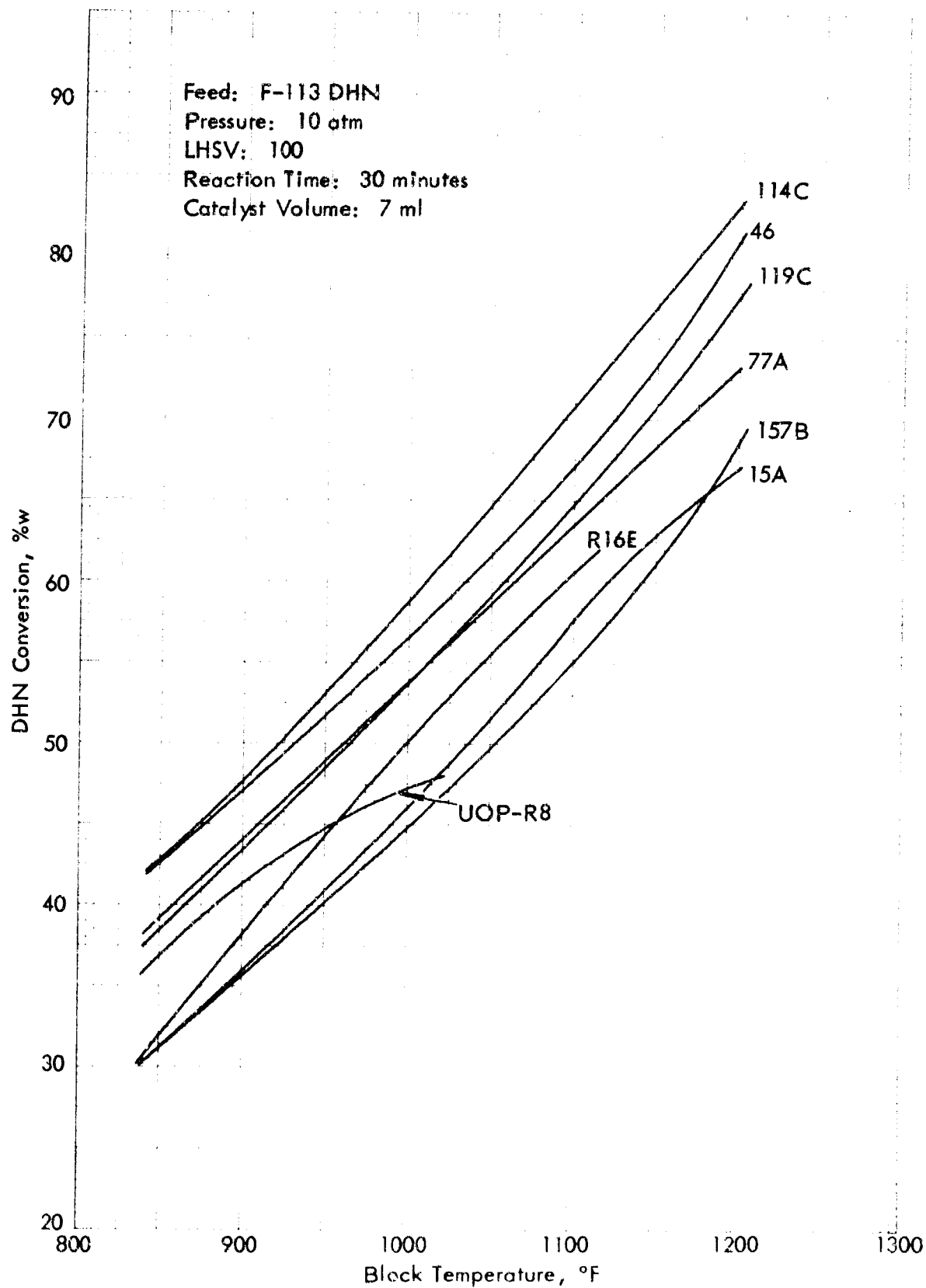


Figure 1. DEHYDROGENATION OF DECALIN: EFFECT OF TEMPERATURE ON CONVERSION

Feed: F-113 DHN  
Pressure: 10 atm  
LHSV: 100  
Reaction Time: 30 minutes  
Catalyst Volume: 7 ml

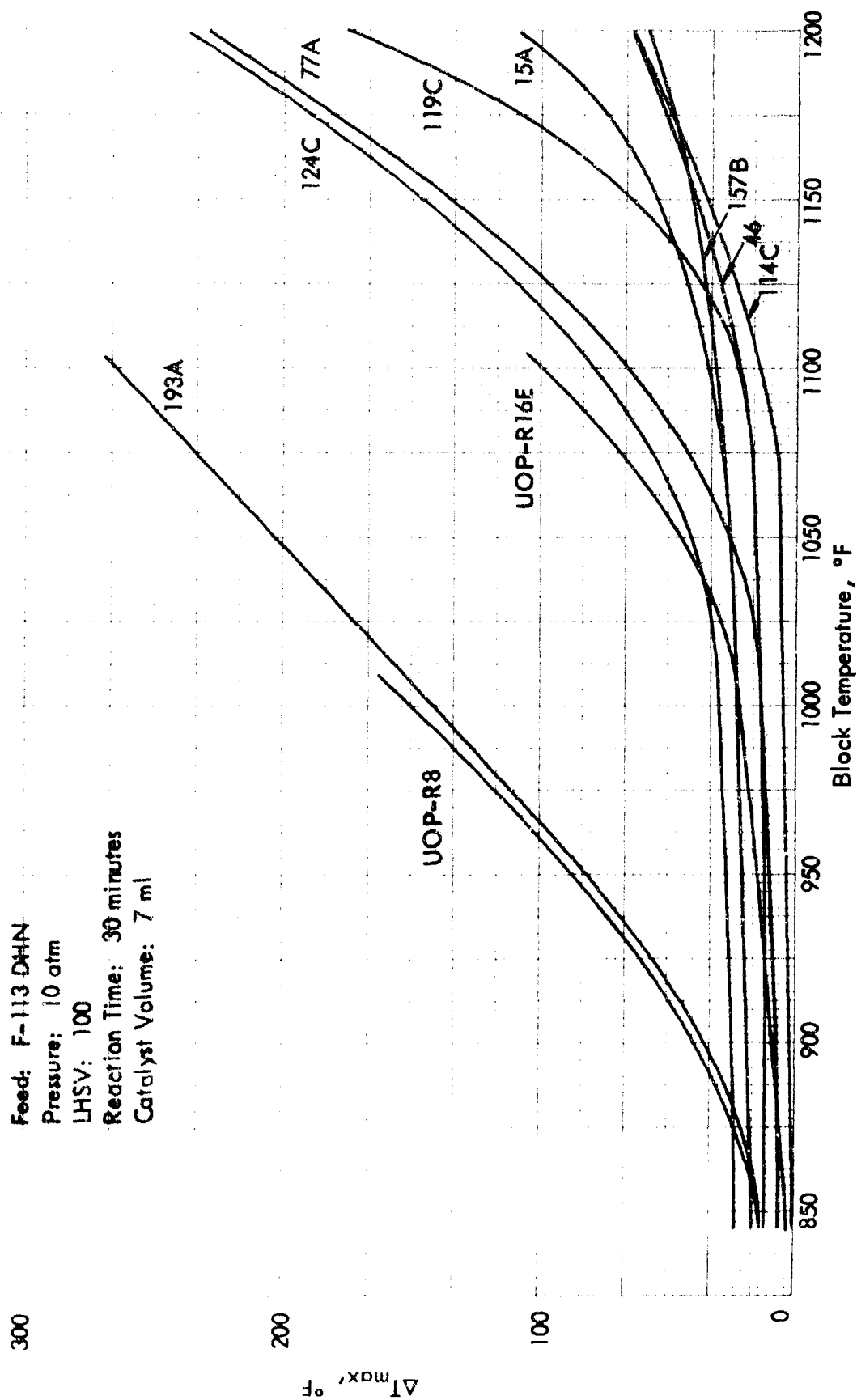


Figure 2. DEHYDROGENATION OF DECALIN: EFFECT OF TEMPERATURE ON CATALYST STABILITY

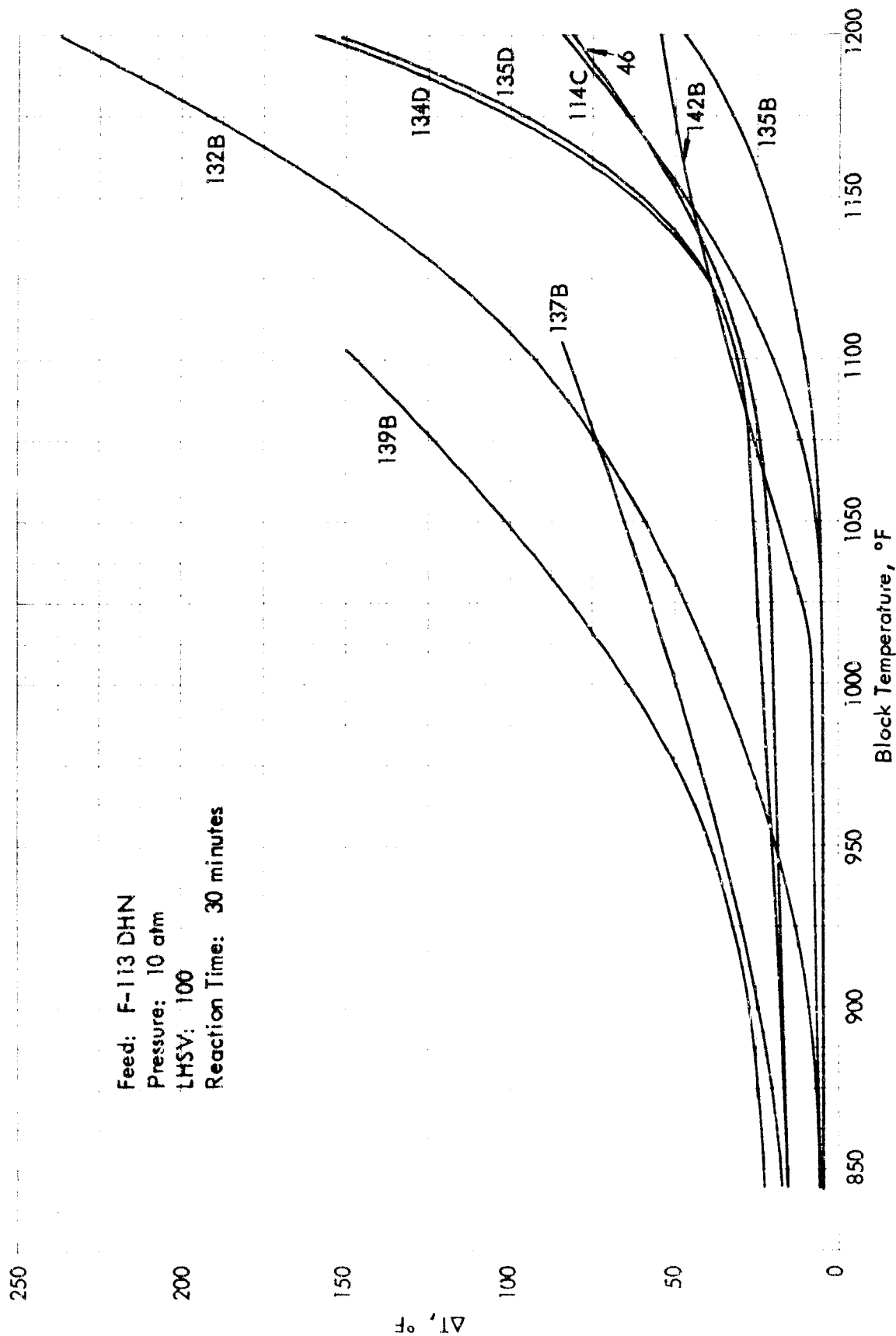


Figure 3. DEHYDROGENATION OF DECALIN: EFFECT OF TEMPERATURE ON CATALYST STABILITY

In earlier work it was shown that stability varied greatly with the catalyst supports.<sup>3)</sup> Further it appeared that the best supports were those with smaller pore diameters. Figure 4 shows the increase in catalyst bed temperature ( $\Delta T_{max}$ ) as a function of average pore diameter of the support for catalysts with similar Pt contents and supports at block temperatures of 1022° and 1202°F. Indeed those catalysts with smaller pores appear to be more stable.

#### Dehydrogenation of Methylcyclohexane

The MCH-catalyst system was considerably more stable than the decalin system. Thus with our standard 1% Pt on  $Al_2O_3$  catalyst good stability was observed with MCH at 10 atm pressure but not at 1 atm<sup>4)</sup>, while with DHN good stability was observed at 30 atm but not at 10 atm pressure.<sup>1)</sup> Consequently in these studies with MCH, stability tests were made at 1 atm pressure. Each catalyst was tested at a single temperature, 852°F, in a series of successive runs with increasing space velocities of 5, 15, 30, 50, 80 and 100 LHSV. The reaction time at each LHSV was 30 minutes. The test was terminated if the catalyst became inactive before reaching LHSV of 100. Fourteen catalysts were evaluated in these tests, four of which had shown good stability with DHN. The complete data are presented in Table 56 in the Appendix, while the pertinent data are summarized in Table 3.

Catalyst deactivation was measured by the magnitude of the increase in catalyst bed temperature during the 30 minute test period. Conversions and catalyst bed temperatures increases at the highest LHSV are shown in Table 3, which also includes two catalysts for comparison that were tested previously (UOP-R8 and UOP-R16E). Conversion as a function of space velocity are shown in Figure 5 for 1% Pt and in Figure 6 for 4% Pt catalysts.

Of the catalysts tested during this period all but five showed little or no catalyst deactivation at the highest space velocity (catalyst bed temperature change was  $0 \pm 2$ , Table 3). This group included platinum mounted on both commercial and Shell prepared supports. Our test was not severe enough or of sufficient duration to differentiate between these ten catalysts; thus, all of them must be considered to have comparable good stability for MCH dehydrogenation. Commercial UOP-R8 appeared to be the least stable, but stability was improved greatly by UOP by adding a "metal activator" (i.e., UOP-R16E).

Activities varied greatly between catalysts, and at LHSV of 100 the most active catalysts were commercial Girdler T-309C and 10860-171E. Six of these catalysts have been tested with decalin. Further testing of the others with this naphthene are planned, as decalin gives a more severe test of catalyst stability.

In summary, we have evaluated many catalysts for stability for the dehydrogenation of naphthenes without added hydrogen. Almost all of the catalysts contained platinum, either alone or with other metals as bimetallic or trimetallic mixtures, mounted on various supports. In the tests with decalin six of the laboratory prepared catalysts showed moderate stability at 1202°F and good stability at lower temperatures. All of these catalysts contained platinum; one was a bimetallic and one was a trimetallic mixture mounted on four different supports. With the less severe tests with MCH nine of the catalysts showed moderate stability at high space velocity. These catalysts will be evaluated further with decalin.

Table 3. DEHYDROGENATION OF METHYLCYCLOHEXANE: SUMMARY OF CATALYST STABILITY TEST DATA AT TERMINATION OF TESTS

Pressure: 1 atm      Block Temperature: 842°F

Catalyst Number	LHSV	MCH Conversion, %	Catalyst Bed Temperature Increase, °F
10280-44 <sup>a)d)</sup>	80	19.8	76
10280-108	100	31.6	+2
10860-132C <sup>d)</sup>	100	30.9	0
10860-135A <sup>d)</sup>	100	32.5	-2
10860-137B <sup>d)</sup>	100	16.1	27
10860-142B <sup>d)</sup>	100	37.3	-2
10860-170A	100	33.3	+2
10860-170B	80	10.7	180
10860-171A	50	10.8	-b)
10860-171B	100	34.9	+2
10860-171C	100	33.2	0
10860-171D	100	34.8	0
10860-171E	100	41.4	+2
10860-196	80	2.9	29 <sup>b)</sup>
UOP-R8 <sup>c)d)</sup>	30	15.3	168 <sup>b)</sup>
UOP-R16 <sup>c)d)</sup>	100	18.5	31 <sup>e)</sup>
Girdler T-309C <sup>c)</sup>	100	44.4	0

a) Standard Catalyst 1% Pt on Al<sub>2</sub>O<sub>3</sub>.

b) Catalyst almost completely deactivated at the end of the test.

c) Commercial catalyst.

d) Also tested with decalin.

e) Cold spot moved down the catalyst bed.

Figures 4, 5 and 6 follow

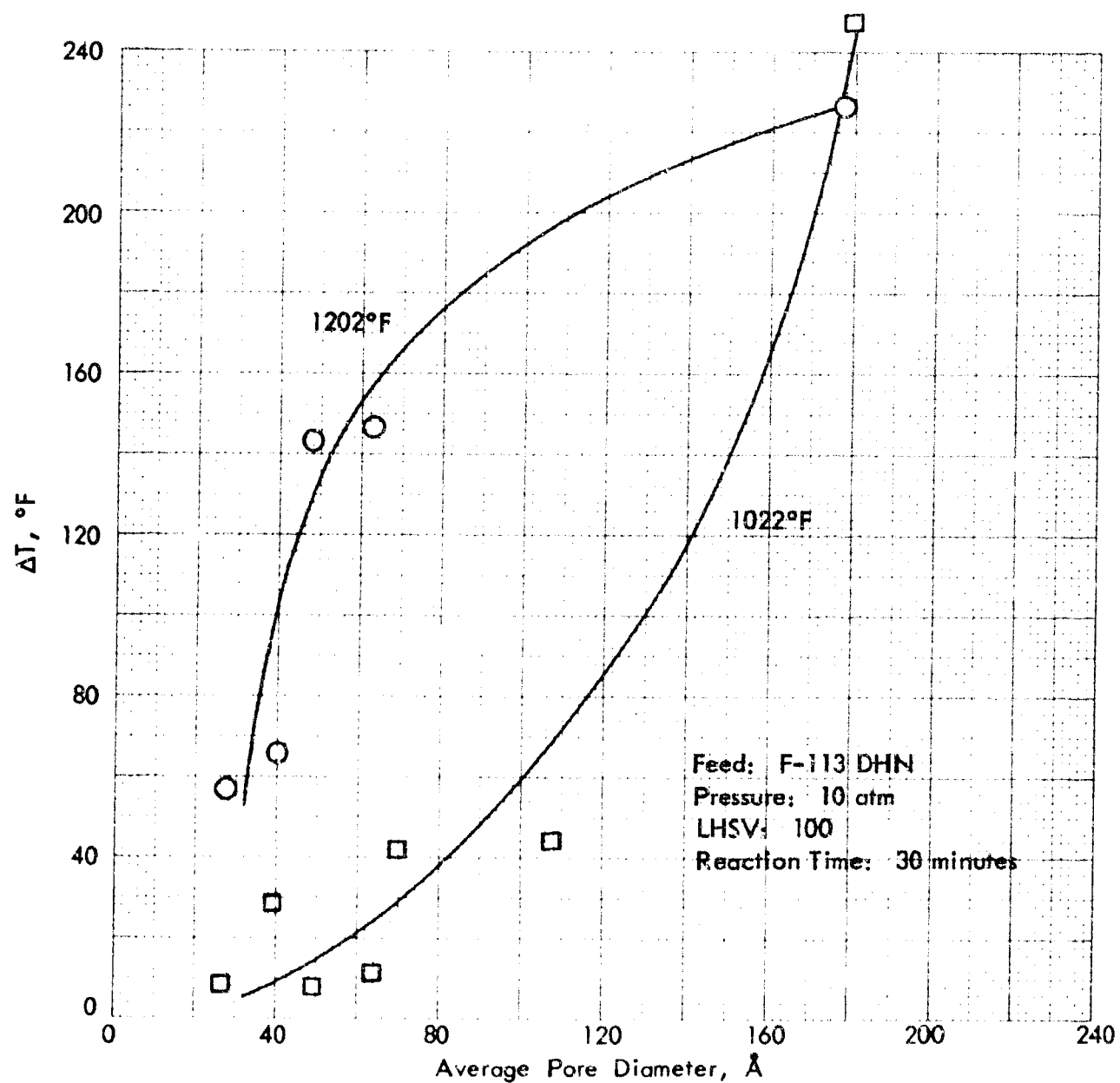


Figure 4. DEHYDROGENATION OF DECALIN: EFFECT OF CATALYST PORE DIAMETER ON STABILITY

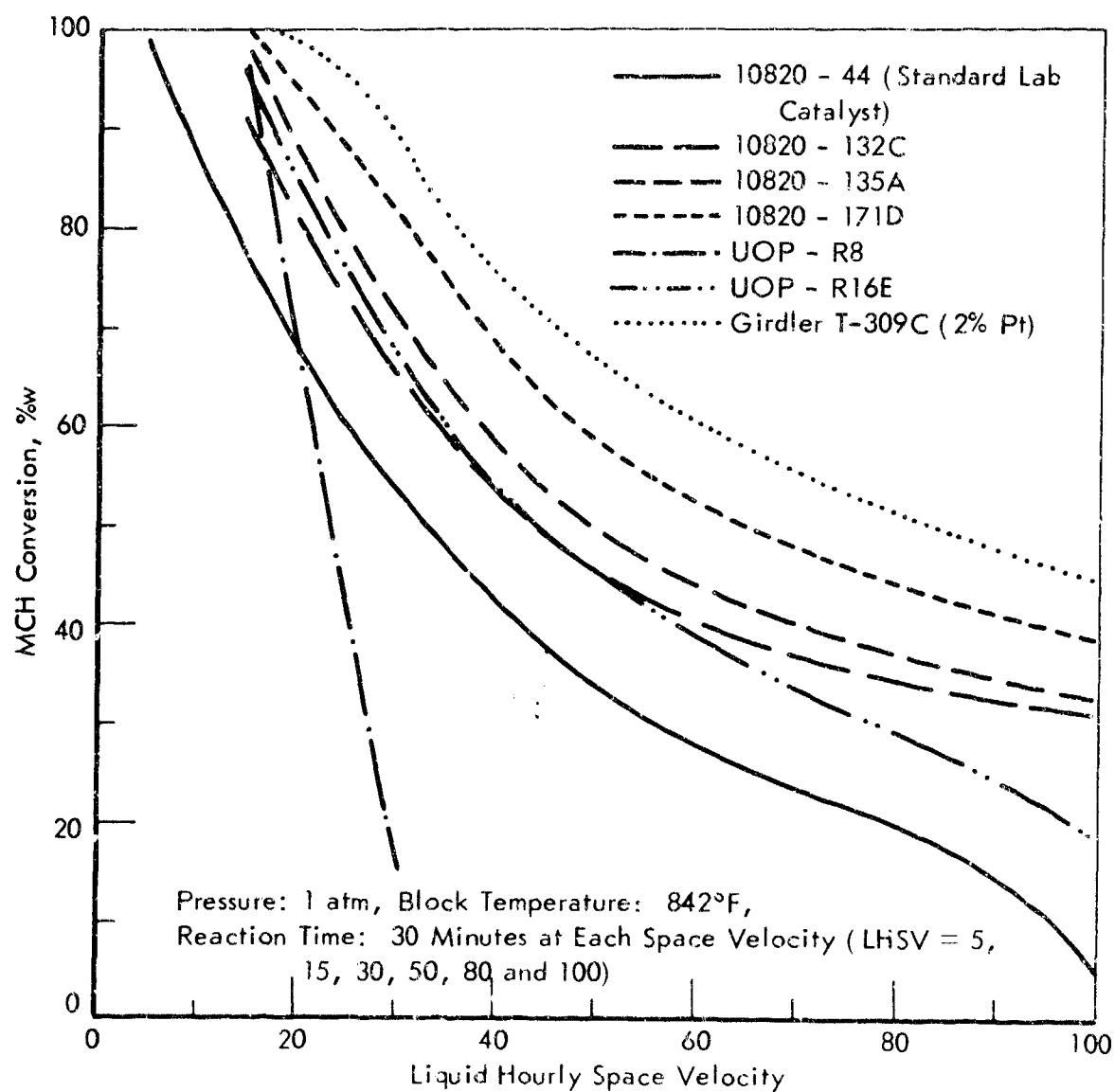


Figure 5. DEHYDROGENATION OF METHYLCYCLOHEXANE OVER ONE PERCENT PLATINUM CATALYSTS

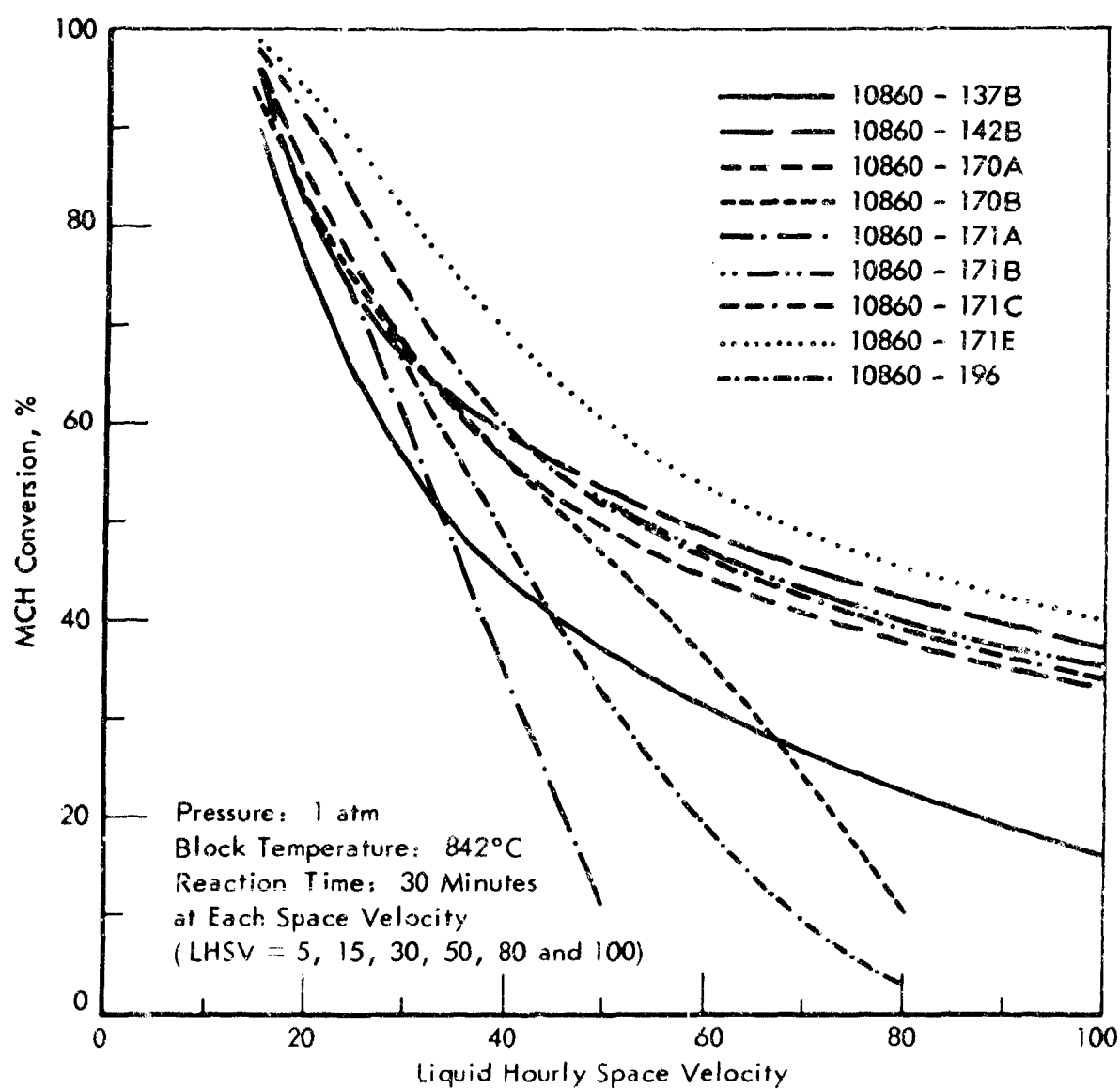


Figure 6. DEHYDROGENATION OF METHYLCYCLOHEXANE OVER  
 FOUR PERCENT PLATINUM CATALYSTS

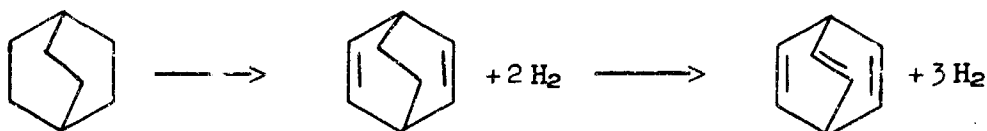


Based on the tests with decalin it appeared that stability was affected by catalyst support composition, physical properties of the catalyst, the catalyst metal content, and composition.

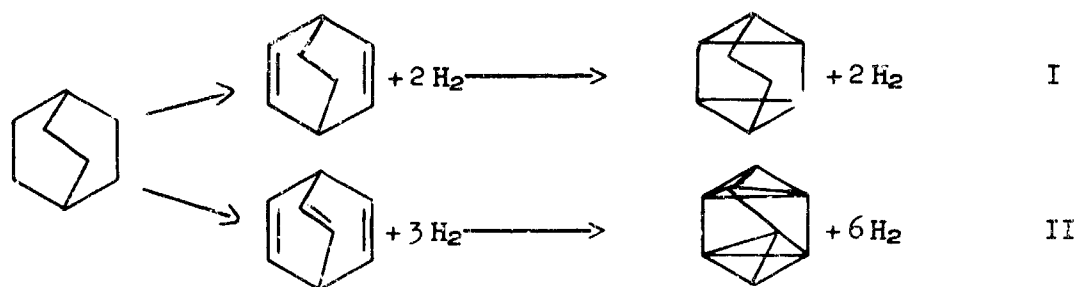
This study is continuing with other supports and metal compositions. Longer tests are being considered in which the catalyst will be run for 50-100 hours at constant reaction conditions.

#### Dehydrogenation of Bicyclo(2,2,2)octane

Bicyclo(2,2,2)octane (BCO) is a monocyclic naphthene with a C-C bridge across the 1,4-position. In principle it can be dehydrogenated to yield three molecules of hydrogen according to the reaction:



The endothermic heat of this reaction is about 1200 Btu/lb for the first step and possibly 1800 Btu/lb for both steps. Another possible reaction of BCO involves small ring formation, thus:



The total endothermic heat for reaction I is estimated at about 2300 Btu/lb and for II over 4000 Btu/lb. Thus BCO is potentially a very attractive fuel, even though it may be difficult to carry out the reaction beyond the first steps.

An exploratory study of the dehydrogenation of BCO was initiated using our pulse reactor. In this system a carrier gas such as helium or hydrogen flowed through the reactor. A small amount of liquid feed (i.e., 1  $\mu$ l) was injected into the gas stream and was carried through the reactor as a "pulse". The exit gas was led directly into a GLC for analysis. The reactor was a 1/4" O.D. stainless steel tube (type 304) five inches long and was heated by an electric furnace. This reactor system is described in detail in the Appendix. GLC analyses were made with an F and M Model 5754 chromatograph using a hydrogen flame detector with a 160' capillary column 0.010" I.D. coated with SF96.

BCO was tested under conditions of both thermal and catalytic reaction at 10 atm pressure. One microliter of liquid feed was injected per pulse. BCO melts at 334°F. Hence, it was dissolved in a solvent in order to inject it into the reactor. Mesitylene (1,3,5-trimethylbenzene, TMB) and n-hexane were used as solvents. Neither one was particularly satisfactory; both were catalytically

reactive and gave reaction products similar to those obtained with BCO. The solubility of BCO in the above solvents was limited to about 30% in TMB and 40% in n-hexane.

Product identification was based on GLC emergence times obtained with pure compounds. With BCO numerous side reactions are possible such as cracking and dealkylation leading to the formation of benzene, toluene, ethyl benzene, p-xylene, and their corresponding naphthenes.

#### Thermal Reaction

The thermal reaction was studied over the temperature range of 572° to 1202°F with both helium and hydrogen carrier gas. Apparent contact times (ACT) were 4.2 to 0.2 seconds. (Apparent contact times were calculated from the carrier gas flow rates and the void volume in the reactor tube. The void volume was assumed to be one-half of the bulk volume of the quartz chips.) The reactor tube was filled with quartz chips having a bulk volume of 2.1 ml. Feed was 25% BCO in mesitylene (TMB). Pure TMB was tested separately. The complete data for TMB are shown in Table 4.

With He carrier gas TMB was reasonably stable, and at contact times of 4.0 and 3.7 seconds, conversions of only 8.0% and 2.2% were observed at 1202°F and 1112°F, respectively (Table 4). Lower conversions were observed at shorter ACT. Based on first order kinetics (see Appendix) the apparent activation energy was 37.1 k. cal/mole (1202° to 1112°F; ACT = 3.7 sec). Figure 7 is an Arrhenius plot of the data.

With H<sub>2</sub> carrier TMB was considerably more reactive than with He at 1202°F but not at 1112°F. Also the apparent activation energy was higher with H<sub>2</sub> carrier. For example with H<sub>2</sub> and an ACT of 3.7 seconds, 24.5% conversion was observed at 1202°F and only 2.4 at 1112°F, which corresponded to an apparent activation energy of 79.2 kcal/mole (Figure 7).

Products and product distributions appeared to be similar with both carrier gases, based on GLC emergence times. With this hydrocarbon the principal reaction product appeared to be ethyl benzene (peak no. 4, Table 4).

BCO was more reactive thermally than TMB by about a factor of 3 to 4, based on first order rate constants. Further, as was observed with TMB, BCO was more reactive with H<sub>2</sub> than He at 1202°F but not at 1112°F. The complete data are given in Table 5, which shows the values for the product analyses in parentheses and the calculated values on a TMB-free basis. These calculated values were also corrected for any contribution to the various components by thermal reaction of TMB, using the data of Table 4. A typical correction calculation is shown in the Appendix. With BCO the lower molecular weight hydrocarbons were the principal reaction products (possibly benzene or toluene).

Activation energies with BCO were 38.3 kcal/mole and 74.2 kcal/mole with He and H<sub>2</sub>, respectively. The data are shown in Figure 7.

Table 4. THERMAL REACTION OF MESITYLENE: PULSE REACTOR

Pressure: 10 atm  
Pulse Volume: 1  $\mu$ l  
Reactor Filled With Quartz Chips.

Run 11023-	106-3	107-1	107-5	108-1	109-3	106-1	106-2	107-2	107-3	107-4	105-2	105-1	104-3
Block Temperature, $^{\circ}$ F	1022	1022	1112	1112	1202	1202	1022	1022	1112	1112	1202	1202	104-3
Carrier Gas	He	He	He	He	He	He	He	He	He	He	He	He	He
Carrier Gas Flow Rate, cc/min	50	200	50	200	50	600	1000	50	50	900	50	200	1000
ACT, seconds	4.2	0.99	4.0	0.99	3.7	0.33	0.19	4.2	4.0	0.99	3.7	0.31	0.19
Product Analysis, %w													
Peak No. 1	0.0	0.0	0.1	0.0	0.3	0.0	0.0	2.9	0.5	0.1	3.8	1.4	0.6
2	0.0	0.0	0.0	0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.1	0.1	0.0
3	0.0	0.0	0.1	0.0	0.5	0.0	0.0	0.4	1.9	0.0	1.4	0.3	0.1
4	0.1	0.0	1.6	0.4	6.7	0.6	0.2	5.2	0.0	0.6	18.7	7.5	2.1
5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.1	0.0
6	0.0	0.2	0.0	0.2	0.0	0.3	0.0	0.0	0.0	0.3	0.1	0.3	0.3
(TMB) 7	99.1	99.0	97.5	98.7	91.7	98.9	99.6	91.5	97.3	98.2	75.3	89.7	96.8
8	0.2	0.2	0.2	0.2	0.2	0.0	0.0	0.0	0.2	0.2	0.3	0.2	0.0
9	0.5	0.5	0.4	0.4	0.4	0.0	0.0	0.0	0.0	0.5	0.2	0.3	0.1
10	0.1	0.1	0.1	0.1	0.1	0.2	0.2	0.0	0.0	0.1	0.0	0.1	0.6
TMB Conversion, %w	0.6	0.7	2.2	1.0	8.0	0.8	0.1	8.2	2.4	1.5	24.5	10.0	2.9
First Order Rate Constant, $\text{sec}^{-1} (\times 10^3)$	-	-	0.67	-	2.13	-	-	-	0.74	-	-	8.67	-

Table 5 and Figure 7 follow

Table 5. THERMAL REACTION OF BICYCLO(2,2,2)OCTANE: PULSE REACTOR

Pressure: 10 atm Reactor Filled with Quartz Chips  
Pulse Volume: 1  $\mu$ l Feed: 23% BCO in Mesitylene

Run No. 11623-	99-1	99-2	100-2	100-3	100-4	102-1	102-2	102-3	102-4	99-4	98-4	100-1	100-5	101-1	101-2	102-5	103-2	104-2
Block Temperature, °F																		
Carrier Gas																		
Carrier Gas Flow Rate, cc/min																		
AUT, seconds																		
Product Analysis, % Peak No. 1																		
2	4.2	1.4	1.2	0.0	0.0	9.3	2.2	0.4	0.0	1.3	0.6	0.4	0.0	0.4	0.4	20.7	2.8	0.9
3	0.4	0.4	3.3	0.4	0.0	12.3	2.7	0.9	0.0	2.6	0.4	0.4	3.0	1.7	0.4	25.0	6.1	1.3
3A	0.4	0.0	0.0	0.0	0.0	0.9	0.0	0.4	0.0	0.4	0.4	0.0	0.0	0.4	0.0	0.0	0.0	0.4
3B	0.4	0.4	0.1	0.0	0.0	0.6	0.1	0.1	0.0	0.1	0.1	0.0	0.1	0.1	0.0	0.0	0.2	0.2
4	0.0	0.0	0.0	0.0	0.0	0.9	0.0	0.0	0.0	0.0	0.8	0.4	0.4	0.4	0.4	1.0	0.9	0.0
BCO	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5	3.1	0.8	3.3	0.0	0.4	0.4	0.0	0.0	0.9	0.0	0.8	0.4	3.0	0.0	0.0	0.0	0.0	0.9
6	94.5	98.4	91.7	99.6	99.6	76.2	94.2	98.3	99.1	95.8	95.9	98.4	93.5	97.0	97.4	92.9	90.0	96.4
7	(24.2)	(23.0)	(22.2)	(23.0)	(23.2)	(17.9)	(21.3)	(22.6)	(22.8)	(22.7)	(23.3)	(23.1)	(21.8)	(22.6)	(22.7)	(11.0)	(19.8)	(21.6)
8	(0.1)	(0.3)	(0.1)	(0.2)	(0.2)	(0.1)	(0.2)	(0.2)	(0.0)	(0.1)	(0.1)	(0.0)	(0.0)	(0.0)	(0.2)	(0.2)	(0.2)	(0.4)
9	(0.0)	(0.0)	(0.0)	(0.0)	(0.0)	(0.0)	(0.0)	(0.0)	(0.0)	(0.2)	(0.2)	(0.2)	(0.0)	(0.0)	(0.0)	(0.0)	(0.0)	(0.0)
10	(74.3)	(75.1)	(73.8)	(75.7)	(76.2)	(70.0)	(75.3)	(76.4)	(77.0)	(75.0)	(75.8)	(75.7)	(75.4)	(75.5)	(76.4)	(66.7)	(72.6)	(75.0)
BCO Conversion, %	(0.2)	(0.0)	(0.1)	(0.2)	(0.0)	(0.1)	(0.2)	(0.0)	(0.0)	(0.1)	(0.2)	(0.1)	(0.2)	(0.1)	(0.0)	(0.2)	(0.1)	(0.0)
First Order Rate Constant, sec <sup>-1</sup> (x 10 <sup>3</sup> )	(0.4)	(0.2)	(0.3)	(0.4)	(0.2)	(0.3)	(0.4)	(0.1)	(0.0)	(0.3)	(0.0)	(0.4)	(0.4)	(0.3)	(0.1)	(0.2)	(0.2)	(0.0)
	(0.1)	(0.1)	(0.1)	(0.1)	(0.1)	(0.1)	(0.1)	(0.0)	(0.0)	(0.1)	(0.1)	(0.1)	(0.1)	(0.1)	(0.0)	(0.1)	(0.1)	(0.0)
	5.5	1.6	8.3	0.4	0.4	23.8	5.8	1.7	0.9	4.2	4.1	1.6	6.4	3.0	2.5	47.1	10.0	3.6
	-	-	2.79	-	-	9.15	-	-	-	-	-	-	2.13	-	-	21.40	-	-

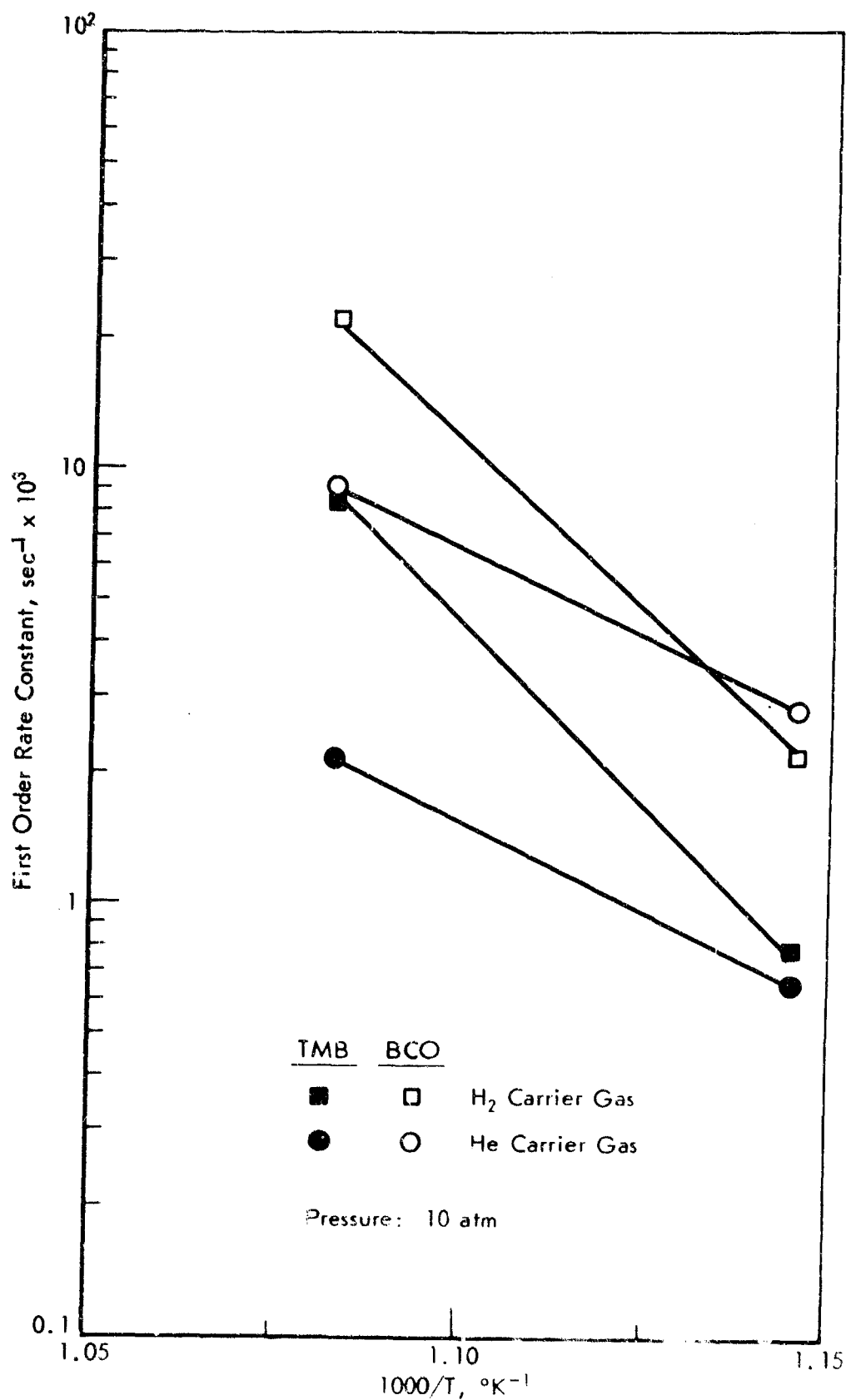


Figure 7. EFFECT OF TEMPERATURE ON THERMAL REACTION OF MESITYLENE AND BICYCLO(2,2,2)OCTANE

The enhanced reactivities of TMB and BCO with H<sub>2</sub> carrier gas could have been due to a hydrocracking-type reaction that occurred at a significant rate at 1202°F but not at lower temperatures. This would explain the higher activation energies with H<sub>2</sub> carrier, as the rate of disappearance of starting material at the higher temperature with H<sub>2</sub> would be due to two (main) reactions, while with He only one reaction occurred (i.e., thermal cracking). This effect of H<sub>2</sub> carrier on naphthene reactivity was observed earlier in work with dimethanodecalin and bicycloheptane.<sup>3)</sup> Based on first order rate constants the reactivity of BCO was less than that of bicycloheptane (BCH) by about a factor of 1.7 to 2.4 (Table 6).

Table 6. COMPARISON OF THERMAL REACTION RATES OF  
BICYCLO(2,2,1)HEPTANE AND BICYCLO(2,2,2)OCTANE:  
PULSE REACTOR

ACT = 4.0 to 3.7 seconds		Pressure: 10 atm.	
Temperature °F	Carrier Gas	First Order Rate Constant, sec <sup>-1</sup> x 10 <sup>3</sup>	
		BCO	BCH <sup>a)</sup>
1112	He	2.8	4.8
	H <sub>2</sub>	2.1	5.0
1202	He	9.2	22.0
	H <sub>2</sub>	21.4	37.0

a) Table 48, pg. 107, reference 3.

#### Catalytic Dehydrogenation

Dehydrogenation of BCO was studied over the temperature range of 572° to 1022°F with both He and H<sub>2</sub> carrier gas. Two feedstocks were used, namely, BCO dissolved in TMB and in n-hexane. Four laboratory catalysts were tested which were 1% Pt on Al<sub>2</sub>O<sub>3</sub> (standard catalyst), 10860-132C, 10860-141A, and 11637-190. In these studies 0.25 ml. of catalyst was diluted with 1.0 ml quartz chips. One ul of liquid feed was injected per pulse.

Two of the catalysts were tested with the BCO in TMB feed (about 30% BCO) at 572-752°F using He carrier gas. These were our standard catalyst and 10860-132C; 132C was a bimetallic platinum catalyst. Liquid hourly space velocities varied from 172 to 686 based on total liquid fed or from 52 to 206 based on BCO fed.

With the standard catalyst (1% Pt), 1% BCO conversion was observed at the highest temperature and lowest space velocity (Table 7). Lower conversions were observed at higher space velocities and lower temperatures. This is shown in Figure 8 which shows conversion as a function of space velocity for each test

Table 7 and Figure 8 follow

Table 7. DEHYDROGENATION OF BICYCLO(2,2,2)OCTANE OVER  
1% Pt ON  $Al_2O_3$ : PULSE REACTOR

Pressure: 10 atm Catalyst No: 10280-44  
Carrier Gas: He Feed: 27-30% BCO in  
Catalyst Volume: 0.25 ml Mesitylene  
Catalyst Wt: 0.2349g Pulse Volume: 1  $\mu$ l  
Catalyst Diluted With 1.0 ml Quartz Chips.

Run 11627-	95-1	91-5	92-1	92-4	92-5	93-1	93-2	93-3	93-4	94-2	94-3	95-2	95-3	96-1
Carrier Flow Rate, cc/min.	150	300	600	150	300	600	150	300	600	300	600	150	300	600
Total LHSV	172	343	686	172	343	686	172	343	686	343	686	172	343	686
BCO LHSV	43	86	172	43	86	172	43	86	172	86	172	43	86	172
Block Temperature, $^{\circ}F$	572			617			662			707			752	
Product Analysis, % (TWG-free) <sup>a)</sup>														
U <sub>2</sub> b)	3.7 (1.0)	3.2 (0.9)	1.9 (0.5)	4.7 (1.4)	3.6 (1.0)	2.6 (0.7)	7.7 (2.4)	5.8 (1.7)	3.8 (1.2)	5.9 (1.7)	4.0 (1.1)	16.6 (6.3)	10.5 (3.2)	9.9 (3.0)
Benzene	2.6 (0.7)	6.1 (1.7)	4.1 (1.1)	7.7 (2.3)	6.0 (1.7)	6.3 (1.7)	12.5 (3.9)	12.7 (3.7)	8.6 (2.7)	13.5 (3.9)	10.3 (2.8)	27.7 (10.5)	22.2 (6.8)	23.6 (7.2)
Toluene	1.2 (0.5)	1.4 (0.4)	0.7 (0.2)	3.3 (0.5)	1.1 (0.3)	1.1 (0.3)	3.5 (1.1)	1.7 (0.5)	1.9 (0.6)	2.1 (0.6)	1.5 (0.4)	8.4 (3.2)	3.6 (1.1)	3.6 (1.1)
Ethyl Benzene plus Bicyclooctene	4.1 (1.1)	3.2 (0.8)	1.9 (0.5)	8.3 (2.4)	4.3 (1.2)	3.3 (0.9)	11.2 (3.5)	7.9 (2.3)	4.8 (1.5)	5.6 (1.6)	5.5 (1.5)	9.5 (3.6)	7.2 (2.2)	6.0 (1.8)
m-p-Xylene	14.8 (4.0)	2.2 (0.6)	3.3 (0.9)	11.7 (3.3)	6.0 (1.7)	4.0 (1.1)	16.0 (5.0)	8.2 (2.4)	5.7 (1.8)	8.3 (2.4)	5.5 (1.5)	26.4 (10.0)	13.4 (4.1)	11.3 (3.4)
o-x)	1.1 (1.1)	3.2 (0.9)	1.5 (0.4)	6.3 (1.9)	4.3 (1.2)	2.6 (0.7)	6.1 (1.9)	4.8 (1.4)	2.9 (0.9)	4.5 (1.3)	2.2 (0.6)	2.4 (0.9)	4.2 (1.3)	2.3 (0.7)
BCO	60.5 (18.5)	80.5 (23.4)	86.7 (23.4)	60.7 (18.2)	74.7 (21.0)	80.1 (21.8)	42.9 (13.4)	58.8 (17.1)	72.4 (22.8)	60.1 (17.3)	71.0 (19.3)	9.0 (3.4)	38.9 (11.9)	43.0 (13.0)
Mesitylene	72.7 (22.7)	70.2 (20.2)	73.0 (21.0)	70.0 (20.0)	71.8 (21.8)	72.8 (21.8)	68.0 (20.0)	70.9 (20.9)	68.3 (20.3)	71.2 (21.2)	72.6 (21.6)	62.0 (18.0)	69.1 (20.1)	69.8 (20.8)
BCO Conversion, %	31.1	19.4	13.3	39.3	25.3	19.9	57.1	41.2	27.6	39.9	29.0	91.0	61.1	57.0
First Order Rate Constant, sec <sup>-1</sup>	0.168	0.195	0.257	0.234	0.274	0.415	0.413	0.520	0.630	0.517	0.697	1.272	0.997	1.779

a) Numbers in parentheses give total product analysis.

b) U<sub>2</sub> = unidentified; emerged before n-hexane.

c) o-x = unidentified; emerged after m-p-Xylene.

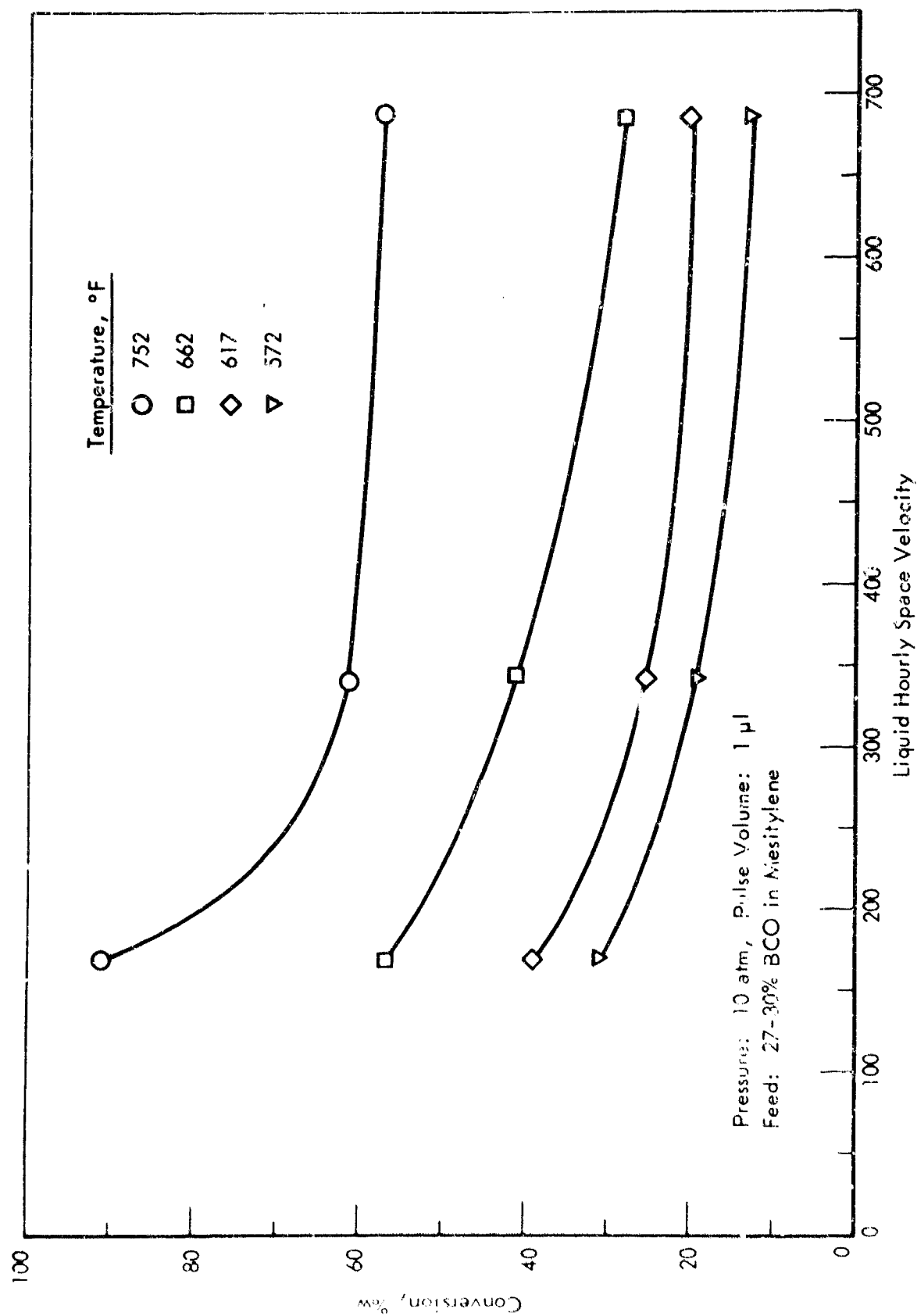


Figure 8. DEHYDROGENATION OF BICYCLO(2,2,2)OCTANE WITH ONE PERCENT PLATINUM ON ALUMINA IN PULSE REACTOR



temperature. There was no measurement of catalyst deactivation during these tests; hence, the conversion values are minimal. The complete data are presented in Table 7 in the order in which the tests were performed. Product analyses, calculated on a TMB-free basis, are shown in the Table 7, and the complete product analyses are shown in parentheses. The calculated values were obtained assuming no TMB reaction. This will introduce a slight error at the higher temperatures, as, in a separate experiment with pure TMB at 752°F (LHSV = 172, He), it was determined that 1.6% TMB was converted to m-xylene (0.8%), toluene (0.4%), and unidentified products. Thus 1.6% should be the maximum error due to TMB reaction, unless there are synergistic effects when TMB is mixed with BCO.

First order rate constants, calculated from conversion of BCO, increased with increasing space velocity. Presumably this effect was due to changes in pulse shape with varying flow rate of carrier gas. Apparent activation energies were calculated from the rate constants at the same space velocity and were 15 to 18 kcal/mole.

Product material was principally benzene and other alkyl aromatics. No dehydrogenation products with bicyclo structures were observed. This suggests that with this catalyst ring opening at one of the secondary carbon atoms is faster than dehydrogenation of the ring, or that the two reactions occur simultaneously.

Based on the first order rate constants the BCO was more reactive than BCH. A comparison of the reactivities of the two naphthenes is shown below:

Table 8. COMPARISON OF REACTIVITIES OF  
BICYCLO(2,2,2)OCTANE AND  
BICYCLO(2,2,1)HEPTANE:  
PULSE REACTOR

<u>Temperature, °F</u>	<u>LHSV</u>	<u>First Order Rate Constant, sec<sup>-1</sup></u>	
		<u>BCO</u>	<u>BCH<sup>a)</sup></u>
1112	43-57	0.413	0.022
	172-238	0.630	0.070

a) Table 51, pg 113, Reference 3.

Preliminary results indicated that considerably more catalyst poisoning occurred with BCH, probably due to formation of cyclopentadiene structures.

Catalyst 132C was more active initially than our standard catalyst, but deactivated appreciably with use. For example, initially 47.6% BCO conversion was observed compared to only 31.1% with our standard catalyst (572°F, LHSV = 172). However, at 752°F (after successive tests at lower temperatures) only 58.4% conversion was observed compared to 91% with the standard catalyst. Finally, on returning to the original test conditions (572°F, LHSV = 172) only 10.1% conversion with 132C was observed. Thus, this latter catalyst had very poor stability for BCO dehydrogenation. The complete data are shown in Table 9.

Table 9 follows

Product distributions were similar to those observed with the standard catalyst, namely benzene and higher aromatic homologues. No component with a bicyclo structure could be definitely identified in the dehydrogenation product.

A series of short tests were made with catalysts 10680-141A (141A) and 11637-190 (190) over the temperature range of 662-1022°F. Both catalysts contained the same type of support; 141A was platinum only and 190 was a binary platinum mixture. The feed was 38% BCO in n-hexane. Tests were made at a single LHSV with He (LHSV = 65) and H<sub>2</sub> (LHSV = 130) carrier gas.

With catalyst 141A BCO was dehydrogenated to bicyclooctene in low yield. Best results were obtained at 932°F with H<sub>2</sub> carrier, where 22% BCO was converted to bicyclooctene. Total BCO conversion was 83.5% which gave a yield of bicyclooctene of 26.4% (Table 10). Lower yields were obtained at lower and higher temperatures and with He carrier gas. The complete data are presented in Table 10.

Besides bicyclooctene the principal reaction products were two unidentified components that emerged after bicyclooctene and before BCO (U<sub>2</sub> and U<sub>3</sub>, Table 10), plus a considerable amount of material that emerged in the C<sub>6</sub> region of the GLC chromatograph. This latter group could not be successfully resolved by our analysis system but appeared to be hexanes, hexenes, and benzene.

The emergence times of component U<sub>2</sub> was about that of p-xylene, but the emergence time for U<sub>3</sub> did not correspond to any of the benzene homologues. Thus, identification of these components is pending.

BCO conversions were about the same with He and H<sub>2</sub> at 752°F and lower, but at higher temperatures (932°-1022°F) higher conversions and yields of bicyclooctene were observed with H<sub>2</sub>.

Catalyst 190 was less active, less selective for bicyclooctene, and for some reason appeared to be completely poisoned by hydrogen at 842°F and lower temperatures. For example, at 932°F BCO conversion was 77.5% with He compared to only 5% with H<sub>2</sub>. Further, at 842°F there was absolutely no reaction with H<sub>2</sub> carrier, although with the He 30.1% conversion was observed. This is interesting as this catalyst was designed primarily for dehydrocyclization of paraffins (i.e., ring closure), in which H<sub>2</sub> is part of the system. The complete data are presented in Table 11.

In summary, bicyclo(2,2,2)octane was catalytically dehydrogenated to bicyclooctene in low yield in pulse reactor tests. The reaction was quite sensitive to catalyst properties and to reaction conditions. With one catalyst bicyclooctene was formed with helium present but not with hydrogen, and with two catalysts containing platinum bicyclooctene was formed with one but not with the other. Present tests indicate that bridged-ring naphthenes can be dehydrogenated but that it may take a highly sophisticated naphthene-catalyst system to effect dehydrogenation to the corresponding dienes or trienes with good selectivity. The work is continuing and other catalysts and BCO solvents are being considered.

Table 9. DEHYDROGENATION OF BICYCLO(2,2,2)OCTANE OVER  
CATALYST 10860-132C: PULSE REACTOR

Pressure: 10 atm Carrier Gas: He  
Feed: 20-25% BCO in Mesitylene Pulse Volume: 1  $\mu$ l  
Catalyst Volume: 0.25 ml Catalyst Diluted With  
Catalyst Weight: 0.2403 g 1.0 ml Quartz Chips

Run No. 11521-	109-1	109-2	110-1	110-3	110-4	111-3	112-5	112-4	113-1	113-3	113-4	114-1	114-2	115-2	115-3	115-4	116-1	116-2	117-1
Carrier Flow Rate, cc/min	100	300	600	150	300	600	150	300	600	150	150	300	600	150	300	600	150	300	600
Time	172	343	686	172	343	686	172	343	686	172	172	343	686	172	343	686	172	343	686
Temp.	43	86	172	43	86	172	43	86	172	43	43	86	172	43	86	172	43	86	172
Flow Temperature, $^{\circ}$ F																			
Product Analysis, % (NBS-free %)																			
100	4.8	2.6	1.4	2.7	2.3	1.0	4.7	2.0	1.0	2.2	5.5	1.9	0.5	5.0	2.5	0.4	0.5	0.0	0.0
100	(1.2)	(0.6)	(0.3)	(0.6)	(0.5)	(0.2)	(1.1)	(0.4)	(0.2)	(0.5)	(1.3)	(0.4)	(0.1)	(1.0)	(0.5)	(0.1)	(0.1)	(0.0)	(0.0)
100	3.2	3.1	2.8	4.0	5.1	3.4	34.5	6.0	2.5	7.1	15.5	5.3	2.6	12.6	8.6	3.5	1.5	0.0	0.0
100	(0.8)	(0.7)	(0.6)	(1.1)	(1.1)	(0.7)	(3.4)	(1.2)	(0.5)	(1.6)	(3.7)	(1.1)	(0.5)	(2.9)	(1.7)	(0.9)	(0.3)	(0.0)	(0.0)
100	1.2	2.3	2.5	0.4	0.5	0.0	0.9	0.5	0.5	0.4	0.8	0.5	0.0	0.9	1.0	0.4	0.5	0.0	0.0
100	(0.3)	(0.2)	(0.1)	(0.1)	(0.1)	(0.0)	(0.2)	(0.1)	(0.1)	(0.1)	(0.2)	(0.1)	(0.0)	(0.2)	(0.2)	(0.1)	(0.1)	(0.0)	(0.0)
100	6.1	3.5	2.4	3.5	3.2	5.4	4.2	6.0	6.6	5.4	5.0	7.7	6.7	5.7	8.1	5.4	4.0	2.7	0.0
100	(1.6)	(0.8)	(0.5)	(0.8)	(0.7)	(1.1)	(1.0)	(1.2)	(1.3)	(1.2)	(1.2)	(1.6)	(1.3)	(1.3)	(1.6)	(1.4)	(0.8)	(0.5)	(0.0)
100	0.8	5.2	2.8	4.0	3.7	2.0	6.0	4.0	3.0	4.9	7.6	7.7	4.6	10.4	11.2	5.4	2.5	1.1	1.1
100	(0.2)	(1.4)	(0.6)	(1.1)	(0.8)	(0.4)	(1.4)	(0.8)	(0.6)	(1.1)	(1.8)	(1.6)	(0.9)	(2.4)	(2.2)	(1.4)	(0.5)	(0.2)	(0.2)
100	0.2	5.6	3.3	3.6	5.1	0.5	6.8	2.0	0.5	4.5	8.4	2.9	0.2	8.3	3.5	0.0	1.0	0.0	0.0
100	(0.1)	(1.5)	(0.7)	(1.8)	(1.1)	(0.1)	(1.6)	(0.4)	(0.1)	(1.0)	(2.0)	(0.6)	(0.1)	(1.9)	(0.7)	(0.0)	(0.2)	(0.0)	(0.0)
100	52.4	77.1	86.7	75.2	80.2	87.3	60.9	79.6	85.9	75.9	51.7	74.0	85.1	15.2	64.6	60.2	89.9	96.3	98.9
100	(11.2)	(17.5)	(16.3)	(17.0)	(17.4)	(17.8)	(14.7)	(16.0)	(17.0)	(17.0)	(12.3)	(15.4)	(16.6)	(10.4)	(12.8)	(15.8)	(17.9)	(18.3)	(18.8)
100	(74.8)	(77.2)	(76.2)	(77.4)	(78.3)	(79.6)	(76.6)	(79.9)	(80.2)	(77.5)	(77.5)	(79.2)	(80.5)	(79.9)	(80.2)	(90.1)	(79.4)	(80.7)	(81.0)
100	47.0	42.3	43.3	24.8	19.8	12.7	37.1	20.4	14.1	24.1	48.3	26.0	14.9	58.4	35.4	59.2	10.9	3.7	1.1
100																			

Analysis for total analysis shown in parentheses.  
Unidentified: analyzed before methylene.  
Unidentified: analyzed after methylene.

Table 10. DEHYDROGENATION OF BICYCLO(2,2,2)OCTANE OVER CATALYST 10860-141A

Pressure: 10 atm  
 Feed: 38% BCO in n-Hexane  
 Catalyst Volume: 0.25 ml  
 Catalyst Wt: 0.0966g  
 Pulse Volume: 1  $\mu$ l  
 Catalyst Diluted With 1.0 ml Quartz Chips

Run No. 11767-	71-1	71-3	72-1	72-2	72-3	73-1	73-2	74-1	74-2
Carrier Gas	He	He	H <sub>2</sub>	He	H <sub>2</sub>	He	H <sub>2</sub>	He	H <sub>2</sub>
Carrier Gas Flow Rate cc/min	150	150	100	150	300	150	300	150	300
LHSV									
Total	172	172	343	172	343	172	343	172	343
BCO	65	65	130	65	130	65	130	65	130
Furnace Temp., °F	662	← 752 →		← 842 →		← 932 →		← 1022 →	
Product Analysis, %w									
C <sub>8</sub> and lighter <sup>a)</sup>	71.0	71.3	71.2	80.4	71.6	86.9	75.7	87.0	85.4
U <sub>1</sub> <sup>b)</sup>	0.0	0.6	0.0	0.5	0.8	0.6 <sup>e)</sup>	1.5	0.4 <sup>e)</sup>	1.7
Bicyclooctene	0.8	1.9	0.8	3.0	4.0	2.4 <sup>e)</sup>	8.4	2.4 <sup>e)</sup>	6.5
U <sub>2</sub> <sup>c)</sup>	1.0	1.2	2.8	1.1	7.5	0.7	6.2	0.8	3.4
U <sub>3</sub> <sup>d)</sup>	0.7	1.4	1.7	1.7	2.5	1.0	1.9	0.9	0.8
BCO	26.5	23.6	23.5	13.3	13.6	8.4	6.3	8.5	2.2
BCO Conversion, %w	31.0	38.7	38.8	65.4	64.6	77.9	83.5	77.6	94.2
BCO Converted to BCO <sup>-</sup>	2.0	4.9	2.0	7.8 <sup>f)</sup>	10.4	6.3 <sup>f)</sup>	22.0	6.3 <sup>f)</sup>	17.0
Yield BCO <sup>-</sup>	6.4	12.8	5.1	12.0	16.0	8.0	26.4	8.1	18.0

a) Hexane, hexene, benzene, cyclohexane, and lighter than C<sub>8</sub>.

b) Emerged before bicyclooctene - unidentified.

c) Unidentified; emerged after bicyclooctene.

d) Unidentified; emerged after U<sub>2</sub>.

e) Two unresolved peaks; one was bicyclooctene, the other may have been bicyclo-octadiene.

f) Assume this component is all bicyclooctene.

Table 11 follows

In this respect it is of interest to test BCO-MCH and BCO-DHN feed systems. Presumably interpretation of product analyses should be simplified as the two pure naphthenes dehydrogenate to toluene and to tetralin and naphthalene over these catalysts. These feeds would have a practical application also as successful dehydrogenation of BCO in DHN in effect would be extending the heat sink range of this latter naphthene.

#### Thermal Cracking of n-Dodecane Using Additives

There is considerable interest as to the maximum amount of heat sink that can be obtained with a paraffinic type jet fuel (JP-7). The latent and sensible heat obtainable from this material is about 1000 Btu/lb when heated to 1300°F. An additional 300 Btu/lb could be obtained by thermally cracking the fuel to about 50% conversion. However, under conventional cracking reaction conditions some coke is produced, which is undesirable. Also, the rate at moderate temperatures is too low. Under the previous contract an investigation was initiated as to the possibility of enhancing the rate of thermal reaction with concurrent reduction in coke make, using free radical initiating fuel additives. This work is being continued under the present contract.

The experiments were done in the pulse reactor, which is described in detail in the Appendix. In this system a stream of carrier gas flowed through the reactor continuously. At the desired time a small amount of feed (ca 1 microliter) was injected into the carrier gas stream and subsequently passed over the catalyst as a pulse. Reaction products, or a slip-stream sample thereof, were led directly into a GLC for analysis.

In these experiments the reactor tube was filled with quartz chips (10-20 mesh). Liquid hourly space velocities were calculated based on the bulk volume of the quartz (i.e., volume of the empty tube), and the apparent contact time (ACT) was calculated based on the void volume in the tube (i.e., one-half the volume of the empty tube). This is close to the actual contact time and is different from our calculation of ACT for catalytic beds, which ignores catalyst volume.

The tests were done at 10 atm pressure and LHSV of 60 using n-dodecane as the test fluid. Helium was used as carrier gas and 1 ml of feed (n-dodecane + additive) was injected per pulse.

In the first series of experiments fourteen additives were tested at 1112°F. The feed was 2%w or less additive in n-dodecane. (Some of the additives were not soluble at this concentration.) The results are tabulated in Table 12.

Some of the additives were effective in increasing the cracking rate and increases in conversions of 40% to 75% were observed (cf 165-7,8,5). Other additives were ineffective or acted as rate inhibitors (cf 165-2,3,13). The additives used were organic compounds containing various functional groups, and it was evident that some of these groups were more effective than others.

The reaction products were lighter than n-dodecane and presumably were cracked material (Table 12). From GLC emergence times the principal component appeared to be a  $C_8$  hydrocarbon (peak No. 1, Table 11) and was not identified

Table 12 follows

Table 11. DEHYDROGENATION OF BICYCLO(2,2,2)OCTANE OVER  
CATALYST 11639-100: PULSE REACTOR

Pressure: 10 atm  
Feed: 38% BCO in n-Hexane  
Catalyst Volume: 0.25 ml  
Catalyst Wt: 0.0986 g  
Pulse Vol: 1 µl  
Catalyst Diluted With 1.0 ml Quartz Chips

Run No. 11767-	64-1	65-1	65-2	65-3	65-4	66-1	66-3	67-1	68-1	68-2	68-3
Carrier Gas	He	H <sub>2</sub>	He	H <sub>2</sub>	He	H <sub>2</sub>	H <sub>2</sub>	He	He	H <sub>2</sub>	He
Carrier Gas Flow Rate, cc/min	150	300	150	300	150	300	300	150	150	300	150
LHSV											
Total BCO	172 65	343 130	172 65	343 130	172 65	343 130	343 130	172 65	172 65	343 130	172 65
Block Temperature, °F	← 662 →	← 75 →	← 842 →	← 842 →	← 842 →	← 842 →	← 842 →	← 842 →	← 932 →	← 932 →	← 1022 →
Product Analysis, %w											
Cs and lighter	66.2	62.8	66.0	63.9	66.2	a)	a)	71.9	71.9	63.2	87.2
Bicyclooctene	1.0	0.0	0.6	0.0	1.2	-	-	1.4	1.1	0.3	0.4
U <sub>1</sub> )	0.2	0.0	0.0	0.0	0.3	-	-	0.0	0.5	0.0	0.7
U <sub>2</sub> )	0.1	0.0	0.3	0.0	2.2	-	-	0.7	1.0	0.1	0.7
BCO	32.7	37.2	33.1	36.1	30.1	-	-	26.7	8.6	36.4	2.0
BCO Conversion, %w	14.6	0.0	13.6	0.0	21.4	a)	a)	30.1	77.5	5.0	94.7
BCO Converted to Bicyclooctene	2.6	-	1.5	-	3.1	-	-	3.7	2.9	0.9	1.0
Yield Bicyclooctene	18.0	-	11.0	-	14.6	-	-	12.3	3.7	17.6	1.1

a) No reaction; only n-hexane and BCO present in chromatogram.

b) Unidentified; emerged after bicyclooctene.

c) Unidentified; emerged after U<sub>1</sub>.

Table 12. THERMAL CRACKING OF N-DODECANE USING ADDITIVES:  
PULSE REACTOR

Pressure: 10 atm  
Pulse Volume: 1 ml  
Reactor Filled With Quartz Chips  
Temperature: 1112°F  
Carrier Gas: He

Carrier Gas Flow Rate:  
200 cc/min  
LEVY: 60  
Contact Time: 1 sec

Run No. 11025-	Feed	116-2	116-1	167-3	167-2	167-1	167-3	168-1	168-4	169-2	169-3	170-1	174-2	174-3	174-4	183-1
Additive 11125-		NONE	165-7	165-10	165-1	165-6	165-8	165-14	165-11	165-4	165-13	165-5	165-9	165-2	165-3	165-2
Additive Concentration, %		-	2	Sat ( $<2\%$ )	Sat ( $<2\%$ )	Sat ( $<2\%$ )	2	2	2	Sat ( $<2\%$ )	Sat ( $<2\%$ )	2	Sat ( $<2\%$ )	Sat ( $<2\%$ )	Sat ( $<2\%$ )	Sat ( $<2\%$ )
Reactor Wall Temperature, °F		1092- 1100	1094- 1103	1094- 1100	1094- 1101	1094- 1101	1092- 1100	1094- 1101	1094- 1101	1098- 1105	1094- 1101	1094- 1101	1094- 1101	1092- 1103	1092- 1100	1100- 1108
Product Analysis, %																
Component No.																
1	0.0	10.7	15.5	11.4	11.4	10.2	20.1	9.7	9.9	11.0	8.1	16.0	10.8	9.0	9.0	10.0
2	0.0	2.3	2.8	2.5	2.5	2.3	3.4	2.1	2.2	2.2	1.7	5.0	2.1	2.1	1.6	2.3
3	0.0	1.7	2.4	1.7	1.7	1.6	3.7	1.8	1.5	1.7	1.4	3.4	1.7	1.6	1.4	1.3
4	0.0	1.4	1.9	1.6	1.6	1.4	2.0	1.4	2.0	1.4	1.2	2.0	1.4	1.3	1.2	1.5
5	0.0	1.2	1.5	1.3	1.3	1.1	2.0	1.2	1.2	1.2	1.0	1.7	1.3	1.1	1.0	1.2
After 5	0.0	0.0	0.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6	0.0	1.0	1.4	0.8	0.8	0.9	1.7	1.0	1.3	1.2	0.9	1.6	1.1	1.0	0.9	1.1
7	0.0	0.3	0.4	0.3	0.3	0.3	0.6	0.3	0.3	0.4	0.4	0.6	0.3	0.3	0.2	0.5
8	0.0	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.2	0.1	0.3	0.1	0.0	0.3	0.3	0.3
9	99.4	80.6	73.2	79.6	79.6	81.4	65.8	82.3	80.5	79.9	83.9	71.6	80.3	83.3	84.4	81.3
10	0.6	0.6	0.3	0.6	0.6	0.6	0.4	0.0	0.9	0.9	1.1	0.0	0.0	0.0	0.0	0.0
Dodecane Conversion, %	--	19.4	26.8	20.4	20.4	18.6	34.2	17.7	19.5	20.0	16.1	38.4	19.7	16.7	15.6	18.7
Increase in Conversions Due to Additives, %	--	--	38.1	5.2	5.2	-4.1	76.3	-8.8	0.5	3.1	-17.0	46.4	1.5	-13.9	-19.6	-3.0

further. In calculating conversion it was assumed that each molecule of dodecane reacted gave one molecule of product; hence, the conversions are maximum values. Product distributions did not appear to be affected by additives, and analyses for several runs with different additives are presented in Table 12. No estimate of coke make could be made from the pulse reactor data.

These results were similar to those observed previously and the maximum rate enhancement was about that observed in the previous work. The best results obtained thus far for all additives tested showed that at 1112°F the rate can be enhanced by about a factor of 1.8 with about 2% additive.

In a second set of experiments a few additives were tested at 1202°F using 3% additive or less in n-dodecane as feed. These additives had been tested previously at 1112°F.<sup>3)</sup> The complete data are tabulated in Table 13, in which the values in parentheses are those for 1112°F.

At 1202°F considerably less enhancement of reaction rate was observed than with the corresponding additives at 1112°F. For example, with 200-1 and 119-13 the overall conversions were increased by 105% and 62%, respectively, compared to increases of 41% and 9% at 1202°F. No essential difference was observed in product material or product distributions at the two temperatures, nor was the product distribution affected by the additives.

Presumably the overall rate of the cracking reaction was due to free radicals generated by (a) thermal means and (b) the additives. As the rate enhancement by the additives declined with increased temperature, this suggests that the activation energies for the production of free radicals by purely thermal means was greater than that for the generation of free radicals by initiators. This work is continuing.

#### Effect of Propane on the Catalytic Dehydrogenation of Methylcyclohexane

The sensible heat sink of the methylcyclohexane (MCH) system could be extended by mixing MCH with a light hydrocarbon such as propane, which would lower the melting point of the system. For additional heat sink MCH would undergo endothermic reaction. Consequently it was of interest to determine if propane affected the reactivity of MCH for dehydrogenation.

A mixture of 30% propane in MCH was tested for MCH dehydrogenation in our bench-scale reactor at 10 atm pressure, 842-1112°F, and an LHSV of 50 (basis MCH fed) over our standard 1% Pt on Al<sub>2</sub>O<sub>3</sub> catalyst. Reaction time was 30 minutes at each temperature. Liquid product material was analysed by GLC and the gaseous products by mass spectrometer. The data are shown in Table 14, which includes data for pure MCH obtained under the same reaction conditions.

Addition of propane enhanced the reactivity of MCH at the lower temperatures but not at 1022°F. This is shown more clearly by Figure 9, which shows conversion as a function of block temperature. This suggests that propane acted as a diluent, and as such improved the heat transfer to the catalyst resulting in higher conversions. At the higher temperatures it appeared that the catalyst was deactivating, possibly due to propane decomposition, although no propylene or lighter hydrocarbons were observed in the gas products. In earlier work with this catalyst and no propane present in the MCH feed, 95% MCH conversion was observed at LHSV of 100, 10 atm pressure, and 1112°F.<sup>5)</sup>

Table 15 and Figure 9 follows



Table 13. THERMAL CRACKING OF N-DODECANE USING ADDITIVES

Pulse Reactor: 1202°F

Pressure: 10 atm  
Pulse Volume: 1 µl  
Reactor Filled With Quartz Chips  
Carrier Gas: He  
Carrier Gas Flow Rate: 200 cc/min  
Contact Time: 1 sec.

Run No. 11325-	161-1	162-1	162-2	162-4	163-2	163-3	164-1	164-3
Additive: 11325-	NONE	199-7	199-12	199-13	NONE	119-21	119-26	200-1
Additive Concentration, %w	--	2.7	2.1	Sat ( $<3.4\%$ )	--	1.7	1.8	2.6
Reactor Wall Temperature, °F	1175-91	1175-91	1175-89	1175-89	1175-91	1177-93	1175-91	1175-91
Product Analysis, %w								
Component No.								
1	41.1	46.2	47.4	45.5	41.9	42.9	44.9	49.8
2	5.7	5.0	6.6	6.4	4.9	4.6	5.9	6.7
3	3.7	6.1	3.9	4.0	3.9	3.9	4.1	4.2
4	2.9	3.1	3.8	3.1	3.0	2.9	3.9	3.0
5	2.2	2.3	1.8	2.1	2.2	2.2	2.5	2.1
After 5	-	-	0.3	-	-	-	0.2	-
6	1.8	1.8	1.1	1.9	2.0	2.0	1.9	1.8
7	0.7	0.6	0.4	0.7	0.8	0.6	0.7	0.7
8	0.2	0.2	0.1	0.2	0.2	0.2	0.2	0.1
9	41.6	34.6	33.4	36.1	41.2	40.7	35.6	31.6
Others	0.0	0.0	1.2	0.0	0.0	0.0	0.0	0.0
Dodecane Conversion, %w	58.4 (20.1) <sup>a</sup>	65.4 (27.6) <sup>a</sup>	66.6 (29.1) <sup>a</sup>	63.9 (32.6) <sup>a</sup>	58.8 (20.1) <sup>a</sup>	59.3 (27.3) <sup>a</sup>	54.6 (35.8) <sup>a</sup>	68.4 (41.2) <sup>a</sup>
Increase in Conversion due to Additive, %	-- (37.3) <sup>a</sup>	12.0 (44.8) <sup>a</sup>	14.0 (62.2) <sup>a</sup>	9.4 --	0.7 (35.8) <sup>a</sup>	1.5 (78.1) <sup>a</sup>	-6.5 (105.0) <sup>a</sup>	17.1

<sup>a</sup> Values obtained previously at 1112°F.

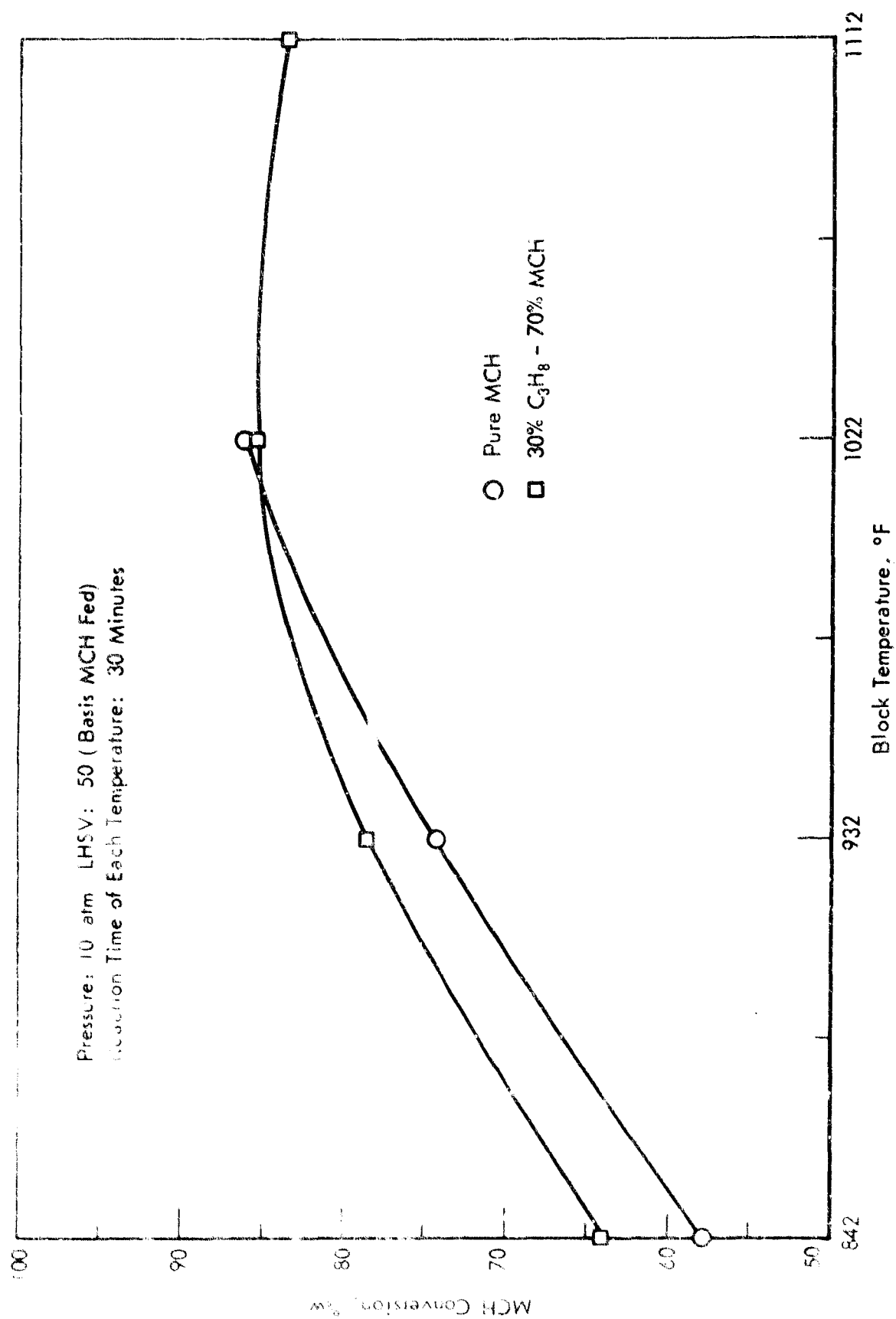


Figure 9. DEHYDROGENATION OF METHYLCYCLOHEXANE: EFFECT OF PROPANE DILUTION

Table 14. DEHYDROGENATION OF PROPANE-METHYLCYCLOHEXANE MIXTURE

Catalyst: 1% Pt on Al<sub>2</sub>O<sub>3</sub> Pressure: 10 atm  
Catalyst Volume: 7 ml LHSV: 50 (basis MCH bed)

Run	11623-			11767-			
	189	190	191	11-1	12-1	12-2	13
Feed	← Pure MCH →			← 30% C <sub>3</sub> H <sub>8</sub> -70% MCH →			
Temperature, °F							
Block	842	932	1022	842	932	1022	1112
Wall	765	837	921	734	806	891	1013
Catalyst Bed	650	694	752	662	671	752	855
Liquid Product Analysis, %							
MCH	42.2	26.1	13.9	36.2	21.6	14.7	16.8
Toluene	57.8	73.9	86.1	63.8	78.4	85.3	83.2
MCH Conversion, %	57.8	73.9	86.1	63.8	78.4	85.3	83.2
Gas Product Analysis, %							
Propan	-	-	-	29.3	26.5	25.5	26.5
H <sub>2</sub>	100	100	100	70.7	73.5	74.5	73.5

## CATALYST STUDIES

We have continued the study and development of conventional granular and coating catalysts and the small scale screening of catalysts for dehydrogenation activity in the micro-test reactor (MICTR), which began under a previous contract.<sup>2)3)</sup> Many additional granular catalysts have been prepared that consist of one or more metals on various supports or mixtures of several unsupported metals from in-house programs. Improvement in dehydrogenation activity at high selectivity still is being sought.

A number of catalysts consisting of 1 and 4% Pt on types 1 or 6 supports with various physical properties have been prepared in furtherance of the concurrent MICTR and bench-scale reactor studies on the activity-stability of dehydrogenation of endothermic fuels, namely MCH and decalin. Included in this study are many additional type 1 supports from both commercial and in-house sources prepared in quantity for bench-scale evaluation. In addition, a study has been started on stabilization of the activity of one of the best type 1 supports, at 1% Pt concentration, by adding single metals at different concentrations. On this support 4% Pt has given better activity-stability than 1% Pt in the bench-scale tests. These metals are expected to suppress the coke forming side reaction that is believed to limit catalyst life during dehydrogenation reactions at low hydrogen partial pressures. Bench-scale testing has shown that greater stability results from using a support with small pore sizes and a high platinum concentration<sup>3)</sup>. Favorable information obtained from this present study probably can be utilized in creating coating formulations with extended catalyst life.

Study has also been initiated in improving the activity-stability of granular platinized type 6 support catalysts, a problem encountered in scale-up preparations, by using two nonalkaline sources of platinum to avoid attack on the surface of the support.

Study has continued of the improvement of catalytic coatings for metal surfaces, such as ease of application, mechanical properties, adherence to metal surfaces, and catalytic activity. New formulations have been studied by metal strip test evaluation and by MICTR tests of candidate coatings in platinized granular form. A possible substitute for the fibrous type 1 component in the best older formulation and which is no longer manufactured has been sought in a widely available natural fibrous material (type 16). Efforts have been made to reformulate coating materials with components shown separately to be activity-stable when platinized. Also, the effect of finer grinding of the wet component mixtures has been studied to obtain better metal adhesion on drying, and to simplify the formulation. Further, formulation with one or more preplatinized components rather than platinization of in situ coatings has been studied as a means of obtaining more exact control of the amount of impregnated platinum.

Through July, 1970, a total of 958 catalysts have been prepared or obtained from proprietary or commercial sources; all of these have been screened in the MICTR during the present and past contracts. Most of the catalysts have been tested in 10-20 mesh particle sizes, including many candidate coating formulations, and one 1/4" OD stainless tubes catalytically coated internally have also been tested. Screening has been for dehydrogenation activity of MCH to toluene without

added hydrogen at 10 atm pressure, 100 LHSV, and 662, 752, and 842°F. The same pump rate (90 ml/hr) was maintained for the coated tubes as for the usual granular catalyst charge (0.9 ml catalyst diluted to 2.0 ml with granular quartz). The coated tubes have been filled with quartz to create better mixing and heat transfer. All catalysts have been compared with reference catalysts 9874-139 or its equivalent 10860-70 (1% Pt/UOP R-8 type  $\text{Al}_2\text{O}_3$ ). The purpose of the screening tests was to obtain a quick comparison with the reference catalyst so as to determine the more promising catalyst compositions, and to eliminate catalysts with activities too low or selectivities too poor to be of practical importance.

A sketch and photographs of the MICTR are shown in Figures 87, 88 and 89 in the Appendix of a previous report<sup>1)</sup> along with a description of the operational details. Subsequent modifications appear in a later report<sup>2)</sup>; more recently the original GLC trace recorder has been replaced with a Westronics recorder. Detailed test data appear in the Appendix, Tables 57 and 58, of this report.

#### Preparation and Evaluation of Granular Catalysts

Most of the particulate catalysts have been prepared by impregnation of various supports (10-20 mesh) with one or more metal salts, followed by drying at 126°C and reduction of the metal (or metals) in situ with hydrogen at 795°F prior to evaluation. Some of the catalysts were unsupported oxide mixtures of various types from several in-house catalytic programs.

Typically, quantities prepared were a few grams for MICTR evaluation with MCH, 30-40 grams for bench-scale studies with endothermic fuels, and 300-400 grams for FSSTR studies (10860-146 and 147). Metal limits were usually between 1 and 4% for the supported catalysts.

Catalysts of the 132, 134, 135, 137, 138, 139, 141, 142, 158, 165, 167, and 161 series were prepared at the 1 and 4% Pt levels on two types of supports with various physical properties. The approximate physical properties of the supports are given in Table 15. These catalysts were prepared primarily for activity-stability studies with decalin in the bench-scale reactor, although they were first screened in the MICTR for activity with MCH (Appendix, Tables 57 and 58). Study of factor affecting activity-stability has been carried out primarily with granular materials, but the factors are considered to be likewise important in catalytic coatings on metals, also.

Catalysts of the 188, 189, 190, 191, and 192 series were prepared from a type 1 support previously shown to be activity-stable in the bench-scale reactor at 4% Pt concentration but much less so at 1% Pt concentration. Various metals (AA-AE) at different concentrations were incorporated into this support in an effort to improve the activity-stability at the lower Pt concentration (1%) and to further investigate stability factors. This treatment has been directed toward the suppression of coke-forming side reactions.

Table 16 summarizes MICTR evaluations of a group of catalysts containing 1 or 4% Pt on type 1, 6, or 10 supports with various physical properties (also Table 57 of the Appendix). Table 17 summarizes MICTR evaluation of additional catalysts prepared from type 1 supports from newer sources. The first group of

Table 15 follows

Table 15. APPROXIMATE PHYSICAL PROPERTIES OF VARIOUS CATALYST SUPPORTS  
(muffled at -1112°F)

Type	Support	Bulk Density (gm/ml)	Surface Area (m <sup>2</sup> / gm)	Pore Vol. (ml / gm)	Av. Pore Dia. (Å)
	Original Configuration				
1 <sup>c)</sup>	Granular	0.50	164	0.719 <sup>g)</sup>	176
"	"	0.58	234	0.384 <sup>a)</sup>	65
"	"	0.76	380	0.51	50
"	"	0.80	230	0.23 <sup>a)</sup>	40
"	"	0.88	210	-0.37	70
"	"	0.75	350	-0.25 <sup>a)</sup>	-28
"	"	0.87	80	0.29 <sup>a)</sup>	145
"c)	"b)	0.63	275	0.62	90
"d)	"	0.65	242	0.494	82
"d)	"	0.61	342	0.367	43
"d)	"	0.87	319	0.323	40
"d)	"	0.60	269	0.526	78
"c)	"	0.49 <sup>e)</sup>	250	0.44	77
"c)	Extrudate	0.68	249	0.55	89
"c)	"	0.63	207	0.66	127
6	Granular	0.72	-800	0.43	-21
"	"	0.38	340	1.15	135
"	Extrudate	0.30	314	1.33	125
10	"	0.52	600	--	-9
16	Granular <sup>f)</sup>	0.41 <sup>f)</sup>	125 <sup>g)</sup>	0.60 <sup>g)</sup>	200 <sup>g)</sup>

a) Does not include macro pore volume.

b) Primary particles are fibrous.

c) Commercial source.

d) In-house source.

e) Bulk density of powder.

f) With 20% type 6 binder.

g) Unbonded powder.

**Table 16. MICTR SCREENING OF VARIOUS PLATINUM SUPPORTED CATALYSTS OF  
DIFFERENT PHYSICAL PROPERTIES**

Conditions: Feed Stock MCH, LHSV 100, 10 atm pressure, no added hydrogen. Temperature variable. GLC samples taken at 5, 8 and 13 minutes, respectively, at each temperature

Cat. No. 10860-	g Pt	Support	MICTR Run No.	wt Charged	Conversion of MCH to Toluene, %		
					662	752	842°F
9874-139	1	UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (Ref.)	1122	0.43	24, 22, 24	50, 46, 47	70, 70, 70
132 A	1	Type 1 Support	1127	0.41	24, 24, 24	52, 49, 49	76, 75, 75
132 B	4	" " "	1118	0.45	24, 22, 24	47, 47, 47	76, 76, 76
134 C	1	" " " a)	1123	0.54	24, 23, 24	58, 54, 55	85, 84, 82
"	"	" " "	1134	0.53	33, 29, 23	53, 53, 53	82, 81, 81
134 D	4	" " "	1124	0.53	20, 24, 21	59, 48, 48	77, 77, 76
"	"	" " "	1135	0.56	(15), 25, 26	54, 53, 49	79, 77, 76
135 C	1	Type 1 Support a)	1125	0.52	25, 26, 26	59, 56, 54	83, 81, 80
"	"	" " "	1137	0.53	27, 25, 27	54, 52, 52	81, 81, 80
135 D	4	" " "	1126	0.57	27, 23, 24	53, 50, 51	78, 77, 77
"	"	" " "	1138	0.54	25, 24, 24	58, 53, 53	83, 81, 81
135 A	1	Type 1 Support a)	1120	0.78	24, 21, 20	51, 48, 49	76, 76, 76
135 B	4	" " "	1121	0.81	25, 22, 22	58, 53, 55	83, 82, 83
137 A	1	Type 1 Support a)	1132	0.73	28, 23, 24	53, 50, 49	79, 78, 77
137 B	4	" " " a)	1139	0.72	26, 30, 27	57, 54, 52	82, 80, 81
144	4	" " " c)	1153	0.83	35, 35, 35	62, 56, 58	87, 87, 86
138 A	1	Type 1 Support a)	1140	0.74	26, 24, 24	51, 49, 49	80, 76, 76
"	"	" " "	1142	0.75	22, 32, 30	55, 53, 53	82, 81, 80
138 B	4	" " "	1141	0.76	28, 25, 31	59, 56, 55	83, 82, 81
"	"	" " "	1145	0.77	33, 33, 28	60, 58, 58	87, 86, 85
139 A	1	Type 1 Support a)d)	1143	0.31	28, 25, 24	44, 44, 42	66, 63, 63
139 B	4	" " "	1144	0.32	26, 30, 27	55, 50, 50	75, 74, 73
141 A	1	Type 1 Support a)	1146	0.31	20, 19, 18	40, 35, 34	56, 55, 55
141 B	4	" " "	1147	0.34	27, 22, 22	49, 45, 44	70, 68, 69
142 A	1	Type 1 Support a)	1149	0.67	23, 23, 22	55, 55, 51	80, 80, 79
142 B	4	" " "	1150	0.72	30, 25, 24	62, 59, 58	89, 88, 88
143 A	1	Type 10 Support b)	1151	0.61	24, 22, 21	38, 36, 41	59, 57, 56
143 B	4	" " "	1152	0.66	25, 24, 21	44, 46, 46	70, 71, 71
147	4	Type 6 Support	1156	0.33	19, 20, 18	44, 41, 41	64, 61, 58
"	4	" " "	1157	0.33	23, 21, 20	43, 41, 41	65, 64, 63
146	4	Type 1 Support e)	1176	0.81	37, 38, 33	60, 58, 59	88, 86, 86
"	"	" " "	1177	0.81	20, 31, 35	63, 59, 59	89, 86, 86
9874-139	1	UOP R-8 type Al <sub>2</sub> O <sub>3</sub> ref.	1136	0.455	22, 24, 21	50, 46, 47	69, 70, 71
10360-136A	1	Type 6	1131	0.586	11, 12, 14	27, 26, 26	41, 49, 57
10360-136B	4	" "	1132	0.598	16, 15, 5	25, 32, 32	48, 47, 48
10860-154A	1	" "	1186	0.638	14, 17, 14	35, 37, 34	47, 41, 40
10860-154B	4	" "	1191	0.657	26, 21, 19	38, 35, 35	50, 45, 45

- a) Muffled before impregnation.  
b) Muffled before Pt (NH<sub>3</sub>)<sub>4</sub> exchange step.  
c) On as received basis.  
d) Surface area 514 m<sup>2</sup>/gm.  
e) Muffled before impregnation.

Table 17. MICR EVALUATION OF PLATINIZED CATALYSTS WITH NEWER TYPE 1 SUPPORTS

Test Conditions: Feed Stock MCH, LHSV 100, 10 atm., no added hydrogen  
(0.9 ml 10-20 mesh catalyst diluted to 2.0 ml volume with quartz)  
GLC Samples taken at 3, 8, and 13 minutes, respectively

Catalyst 10860-	% Pt	Source of Type 1 Support	MICR Run No.	wt. charged, g.	Average Conversion of MCH to Toluene, %		
					662	752	842 <sup>a</sup>
70 (ref)	1	--	1201	0.410	22	46	71
158A <sup>a</sup>	1	commercial	1206	0.535	25	53	83
158B <sup>a</sup>	2	"	1207	0.542	24	52	81
158C <sup>a</sup>	4	"	1208	0.567	24	53	83
165A <sup>a,b</sup>	1	"	1219	0.534	26	56	81
165B <sup>a,b</sup>	4	"	1218	0.549	29	51	80
157A	1	"in house prepn."	1202	0.562	22	50	78
157E <sup>b</sup>	4	"	1203	0.596	25	58	83
170B <sup>b</sup>	4	"	1231	0.597	22	51	80
157B	1	"	1204	0.545	22	52	80
157F <sup>b</sup>	4	"	1205	0.562	27	58	87
170A <sup>b</sup>	4	"	1236	0.559	24	57	87
157C	1	"	1210	0.843	18	36	49
157G	4	"	1211	0.828	18	35	47
157D	1	"	1212	0.527	21	46	74
157H <sup>b</sup>	4	"	1213	0.548	23	50	77
171C <sup>b</sup>	4	"	1237	0.541	27	55	82
171A <sup>b</sup>	4	commercial	1233	0.606	25	57	84
171B <sup>b</sup>	4	"	1234	0.498	25	51	77
171D <sup>b</sup>	1	"	1238	0.712	21	48	69
171F <sup>b</sup>	2	"	1240	0.762	20	48	75
171E <sup>b</sup>	4	"	1239	0.747	22	52	82

a) Granules prepared from fibrous type 1 support.

b) Prepared in larger quantities for bench-scale activity-stability tests with endothermic fuels.



catalysts were predominantly larger scale preparations of various types, mostly with high activity, and were prepared primarily to further the study of activity-stability in the bench-scale reactor with endothermic fuels.

Two general problems of scale-up were encountered with type 6 supports:

(1) High density type 6 support particles decrepitated extensively to smaller than 20 mesh on impregnation with platinous tetrammine dihydroxide, notwithstanding the prehumidification of the particles with water (10860-136A and 136B, Table 16). This was avoided in subsequent preparations (10860-134A and 134B) by a modified technique. Catalysts from this support have been shown in the past to be less active in the MICTR test and less stable in the bench-scale test with decalin than those prepared from low density type 6 support which does not decrepitate on contact with liquid water.

(2) Scale-up of type 6 supported preparations requires longer drying time and thus longer contact time, resulting from thicker catalyst layers during evaporative concentration of the highly alkaline platinum solution. This undoubtedly leads to greater attack of the siliceous surface with consequent dissolution of silica at the expense of surface area contributed largely by the walls of small pores. A glass-like film has been observed at the evaporating interface in the porcelain dish during concentration of impregnating solutions. The experimental preparations designed to avoid this problem are described below.

Earlier study with small batches of catalysts consisting of 1 or 4% Pt on low density type 6 support showed good activity for MCH dehydrogenation in the MICTR, notwithstanding the low charging weight (Table 18, catalysts 10860-114B and 114C). Also, catalyst 114C showed excellent activity-stability on bench-scale tests with decalin. Reproduction on a larger scale gave catalysts less active for MCH dehydrogenation (10860-141A and 141B), and catalyst 141B showed poorer activity with decalin in the bench-scale test than catalyst 114C.

Various factors have been studied to elucidate further this difficulty. These results indicate that when platinum solution 30 is used as a source of platinum equally active catalysts are obtained regardless of whether the low density type 6 support is used on a "as received basis", or is muffled in air at 1112°F, (catalysts 10860-149A and 149B). Slightly higher activity is obtained if the platinum solution is first neutralized (catalysts 10860-149C, 149D, and 166A). This modification of the impregnating solution is expected to play a more important role in activity-stability with decalin in the bench-scale test, because surface attack on the type 6 support by the highly alkaline platinum solution probably damages the catalytic surface more than the neutralized platinum solution. Catalysts of equal performance to those prepared by the latter solution have been obtained when an acidic source of platinum is used (No. 31). The data shown in Table 18 indicate that highly active catalysts can be prepared from extruded low density type 6 support with good correlation of activity with platinum content (10860-128A, 128B, and 128C).

Catalyst 10860-113 (4% Pt, 9-16 mesh, spherical type 1 support), made in large quantity for FSSTR studies, was found to be less active in the MICTR with MCH than earlier counterparts (10280-107B and 91A). These latter catalysts had

Table 18. MICTR EVALUATION AND COMPARISON OF VARIOUS PLATINUM PROMOTED TYPE 6 SUPPORTED CATALYSTS

Conditions: MCH, IHSV 100, 10 atm pressure, no added H<sub>2</sub>, temperature variable.  
(10-20 mesh particle size catalyst)

Catalyst No.	% Pt	Pt Source	Form of Type 6 Support	MICTR Run No.	Average Conversion of MCH to Toluene, %	
					662	752
9874-139	1	30	reference (spherical Al <sub>2</sub> O <sub>3</sub> )	1116	25	48
10860-114B	1	30	granular (low density)	1112	17	38
" 114C	4	30	"	1113	23	52
" 141A	1	30	"	1146	19	35
" 141B	4	30	"	1147	22	45
" 70	1	30	reference (spherical Al <sub>2</sub> O <sub>3</sub> )	1166	24	45
" 149A	4	30	granular <sup>a)</sup> (low density)	1163	24	44
" 149B	4	30	" <sup>b)</sup>	1172	28	43
" 149C	4	30 <sup>c)</sup>	" <sup>a)</sup>	1173	24	47
" 149D	4	30 <sup>c)</sup>	" <sup>b)</sup>	1179	20	46
" 149E	4	31	" <sup>a)</sup>	1174	25	45
" 149F	4	31	" <sup>b)</sup>	1175	24	48
9874-139	1	30	reference (spherical Al <sub>2</sub> O <sub>3</sub> )	1105	25	47
10860-128A	1	30	extruded, granular <sup>d)</sup> (low density)	1098	20	44
" 128B	2	30	"	1106	21	47
" 128C	4	30	"	1100	26	51
" 166A	4	30 <sup>c)</sup>	granular <sup>b)</sup> (low density)	1220	22	43
" 166B	4	31	"	1221	21	43

a) Support as received.

b) Support muffled.

c) Neutralized impregnating solution.

d) Support muffled in air.

been prepared on the same support screened to 10-16 mesh, (Table 20, runs 1108 vs 1155 and 401). A 10-16 mesh portion of catalyst 113 had higher activity (run 1114) but less so than earlier counterparts; thus the particle size range was only partly responsible. Heating of the 9-16 mesh particles of catalyst 113 in air at 932°F improved the activity somewhat (run 1130).

A number of type 1 supported catalysts containing different amounts of metal G at a 1:1 weight ratio with platinum have been tested with MCH in the MICTR without added hydrogen. Suitable control catalysts with the same amounts of platinum without metal G on the same supports have also been tested (Table 19). Under present MICTR conditions the addition of metal G produced no appreciable improvement in activity for dehydrogenation of MCH. Platinum-metal G promoted catalysts have been examined for activity-stability with decalin in the bench-scale test apparatus and the results are given earlier in this report.

Table 19. EFFECT OF METAL G ON PERFORMANCE OF PLATINUM TYPE 1 SUPPORTED CATALYSTS WITH METHYLCYCLOHEXANE IN MICTR

Conditions: LHSV 100, 10 atm. pressure, no added H<sub>2</sub>, temperature variable; 9874-139 reference catalyst.

Catalyst No.	Metals		MICTR Run No.	Average Conversion of MCH to Toluene, %w		
	% Pt	% G		662	752	842°F
9874-139	1	0	1105	26	47	72
10860-129F	0.5	1	1110	23	52	82
" 129A	0.5	0.5	1102	26	54	79
" 129G	1	0	1111	24	54	84
" 129B	1	1	1103	27	53	78
" 132C	1	1	1119	21	49	74
" 129C	2	2	1115	23	51	80
" 129E	4	0	1109	25	55	83
" 129D	4	4	1107	25	56	87
9874-139	1	0	1122	23	47	70
10860-134A	0.5	0.5	1128	26	42	65
" 134B	1	1	1129	21	43	65

Table 21 shows the MICTR evaluations with MCH of 1% platinized type 1 supports containing various amounts of several metals intended to improve activity-stability on more rigorous bench scale testing. Metals AA and AB with content increasing from 1 to 4% decreases initial activity only slightly at 752°F (runs 1263-1259). Metal AB produces a much more pronounced decline of initial activity

Table 20. RELATIVE ACTIVITIES OF CATALYSTS PREPARED FROM SPHERICAL  
TYPE 1 SUPPORT WITH METHYLCYCLOHEXANE IN MICR

Conditions: Same as for Table 18

Catalyst No.	% Pt	Mesh Size of Spherical Particles	MICR Run No.	Average Conversion of MCH to Toluene, %		
				662	752	842°F
10860-70 <sup>a)</sup>	1	10	1148	22	46	68
10280-91B	2	10-16	400	25	52	74
" 91A	4	"	401	26	57	79
" 130G	4	" <sup>b)</sup>	543	23	45	70
10280-107B	4	10-16	1155	22	49	73
" 113	4	9-16	1108	20	43	69
" "	4	10-16	1114	20	46	69
10860-137C	4	9-16 <sup>c)</sup>	1130	21	47	72

a) Reference catalyst on UOP R-8 type  $Al_2O_3$ .

b) Catalyst 10280-91A muffled in air 2 hours.

c) Catalyst 10280-113 muffled in air 1/2 hour.

Table 21. MICTR EVALUATION OF PLATINUM/TYPE 1 S REPORTS CONTAINING SEVERAL CONCENTRATIONS OF VARIOUS METALS INTENDED TO IMPROVE ACTIVITY-STABILITY WITH ENDOTHERMIC FUELS

Test Conditions: Feed stock MCH, LHSV 100, 10 atm., no added hydrogen  
(0.9 ml 10-20 mesh catalyst diluted to 2.0 ml volume with quartz)  
GLC samples taken at 3, 8, and 13 minutes, resp.

Catalyst 10860-	% Pt	% And Type Of Second Metal	MICTR Run No.	wt. Charged g	Average Conversion of MCH to Toluene, %		
					662	752	842°F
70 188A	1	Ref. Control <sup>a)</sup>	1254	0.419	20	45	69
	1		1256	0.303	27	52	81
190A 190B 190C	1	1 AA	1263	0.711	24	54	81
	1	2.3 AA	1265	0.767	23	51	81
	1	4 AA	1266	0.764	20	47	76
188B 188C 188D	1	1 AB	1257	0.738	24	55	84
	1	2.3 AB	1258	0.713	23	51	79
	1	4 AB	1259	0.752	16	45	68
189A 189B 189C	1	1 AC	1260	0.759	29	51	81
	1	2.3 AC	1261	0.731	19	49	68
	1	4 AC	1262	0.747	15	38	47
191A 191B 191C	1	1 AD	1267	0.767	28	54	82
	1	2.3 AD	1268	0.746	21	53	84
	1	4 AD	1269	0.742	22	53	73
192A 192B 192C	1	1 AE	1270	0.736	24	57	87
	1	2.3 AE	1271	0.737	25	53	83
	1	4 AE	1272	0.769	23	50	79

a) For series 10860-188, 189, 190, 191, and 192.

with increasing content (runs 1260-1262). Metal AD at different concentrations has no appreciable effect on initial activity (runs 1267-1269). Metal AE with increasing concentration decreases initial activity only slightly at 752°F, acting in a manner similar to metals AA and AB (runs 1270-1272).

### Preparation of Catalyst Coatings on Metal Surfaces

#### Stainless Steel

A large number of new candidate metal-coating formulations have been studied. Most of these have been made by wet grinding the ingredients with a mortar and pestle. However, in certain cases ball-milling the ingredients together has shown to have certain advantages. Small portions of each candidate formulation were spread as thin coatings, usually 3-7 mils thick, onto the smooth and sandblasted sides of degreased stainless steel strips, 1/2 x 2", or on aluminized stainless strips. These coatings were dried, calcined, and then evaluated for coating thickness, metal adhesion, and physical condition. A few coatings were stored wet in a humid desiccator and then subjected to similar tests to check their storability. A portion of each original formulation was dried and muffled, and the 10-20 mesh particle size platinized and evaluated in the MICTR with MCH. Activity at 752°F is shown in Tables 22 and 23 with coating evaluations. Complete MICTR test data are given in Tables 57 and 58 of the Appendix.

In the past, the more successful thixotropic candidate materials were applied to metal surfaces with freedom from cracks and with good adhesion after drying and muffling, and were platinized to produce active surfaces<sup>3</sup>. It was found that application of the hydrous formulations must be made more swiftly, however, since these formulations dry out rapidly at room temperature while being applied to flat metal surfaces. Coating of tubes internally is less of a problem since the drying proceeds at a slower rate. It has been found that this difficulty can be avoided by the addition of 10 or 20% (based on dry solids) of deliquescent salt No. 22. On drying and thermal decomposition this salt becomes a binder itself, and also within certain limits can supplant part of the No. 6 binder currently used. Examples of catalysts using No. 22 binder alone are the 10860-119A-D series impregnated with 2 or 3% platinum (runs 1084 to 1087) and tube No. 28 (run 1088, Table 57 of the Appendix). While activity of the solid catalysts is high, this type of formulation has poor metal adherence and self strips easily. This apparently accounts for the low activity of 1/4" O.D. coated tube No. 28 (run 1088). Coatings were made with 15% No. 6 binder - 5% No. 22 binder and 10% No. 6 binder - 10% No. 22 binder, which have been used to prepare the active platinized catalysts of series 125 and 126 (runs 1094 and 1101). Metal adhesion of the formulations appears satisfactory. Demonstration of activity of a platinized coated tube was not deemed necessary.

Some experimentation was carried out in which alkaline platinum solutions themselves were used as binders for a powdered support with a spherical particle shape (10860-121 and 123, runs 1091-1092). Although active catalysts resulted on platinization, adhesive properties of this type of formulation were poor.

Tables 22 and 23 follow

Table 22. MICTR EVALUATION AND METAL ADHESIVE PROPERTIES OF  
VARIOUS WALL COATING CANDIDATES

Test Conditions: Feed stock MCH, LHSV 100, 10 a<sup>+</sup>n, no added hydrogen.  
(0.9 ml 10-20 mesh catalyst diluted to 2.0 ml volume with quartz).  
GLC samples taken at 3, 8, and 13 minutes, respectively.

Catalyst 10860-	% Pt	Material Composition	Thickness (mils)	Muffled (°F)	Adhesion		MICTR Run No.	Conv. of MCH To Toluene, 750°F
					Rating	% off		
119A	2	40% particulate type 1 support	13-14 SM	932	poor	a)	1084	52
119B	3	40% fibrous type 1 support 20% type 22 binder (deliquescent before muffling)	" " SB	"	"	a)	1085	59
119C	2	45% particulate type 1 support	---	--	poor	a)	1086	53
119D	3	45% fibrous type 1 support 10% type 22 binder (deliquescent before muffling)	---	1004	"	90	1087	57
125A	2	40% particulate type 1 support	3-6 SM	1004	poor	30	1094	51
125B	3	40% fibrous type 1 support 15% type 6 binder 5% type 22 binder (deliquescent before muffling)	5-15 SB	"	good- excellent	15	1095	49
126A	2	40% particulate type 1 support	---	1004	Excellent	10	1101	58
126B	3	40% fibrous type 1 support 10% type 6 binder 10% type 22 binder (deliquescent before muffling)					1097	57
121 123	2 2 d)	type 1 (spherical particle shape) <sup>c)</sup>	2 2 SB	824 "	poor <sup>b)</sup>	-	1091 1092	55 53

- a) Self-stripped on dehydration.  
b) Easily rubbed off (continuous film).  
c) Platinized.  
d) Neutralized impregnating solution.

Table 23. MICR EVALUATION AND METAL ADHESIVE PROPERTIES OF VARIOUS PREPLATINIZED WALL COATING CANDIDATES

Test Conditions: Feed Stock MCH, LHSV 100, 10 atm., no added hydrogen  
(0.9 ml 10-20 mesh catalyst diluted to 2.0 ml volume with quartz)  
GLC samples taken at 3, 8, and 13 minutes, respectively.

Catalyst 10860-	Pt	Support Composition	SS Adhesive Properties of Support		MICR Run No.	Conv. of MCH to Fluene, %
			Thickness	% Stripped		
70	1	UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	--	--	1194	45
163A	3.2	80% type 1 fibrous support <sup>c)</sup> - 20% type 6 binder	3-4 mils SM 2-3 mils SB	60 20	1197	47
161A	3.2	80% type 1 particulate support <sup>c)</sup> - 20% type 6 binder	4-5 mils SM 2 mils SB	70 35	1198	55
163C	1.6	40% type 1 support (1) <sup>c)</sup> - 40% type 1 support (2) - 20% type 6 binder	5 mils SM 3-7 mils SB	90 40	1194	45
168	1.6	40% type 1 support (1) <sup>c)</sup> - 40% type 1 support (2) <sup>c)</sup> - 20% type 6 binder	2-3 mils SM 1 mils SB	90 50	1197	47
161B	1.6	" " " " " "	3-4 mils SM 2-3 mils SB	95 5	1200	46
163B	3.2	40% type 1 support (1) <sup>c)</sup> - 40% type 1 support (2) <sup>c)</sup> - 20% SiO <sub>2</sub>	3-5 mils SM 3-5 mils SB	45 10	1194	50
160A	1	80% type 16 fibrous support - 20% type 6 binder <sup>d)</sup>	3-7 mils SM	50	1196	47
160B	2	" " " " " "	5-6 mils SB	25	1197	47
160C	4	" " " " " "	" " " " " "	" " " " " "	1198	55
167C	3.2	80% type 16 fibrous support <sup>c)</sup> - 20% type 6 binder	6 mils SM 4 mils SB	30 30	1225	43
161A	3.2	80% type 1 particulate support <sup>c)</sup> - 20% type 6 binder	4-5 mils SM 2 mils SB	70 35	1199	45
172	1.6	40% type 16 support <sup>e)</sup> - 40% type 1 support <sup>c)</sup> - 20% type 6 binder	--	--	1230	47
173	1.6	40% type 16 support <sup>f)</sup> - 40% type 1 support <sup>c)</sup> - 20% type 6 binder	3-4 mils SM 4 mils SB	40 5	1235	51
175	3.2	40% type 16 support <sup>c,f)</sup> - 40% type 1 support <sup>c)</sup> - 20% type 6 binder	4 mils SM 3-4 mils SB	70 35g)	1242	50

SM = Smooth degreased stainless steel.  
SB = Sand blasted stainless steel.  
SS = Stainless Steel.

a) % stripped off when pressure sensitive tape pressed onto surface, and then gently pulled off.  
b) Self stripped during drying.  
c) Pre-platinized with 4% Pt before formulation.  
d) Entire support platinized.  
e) Unmuffled.  
f) Muffled.  
g) On aluminized SS, 2-3 mils thick; 60% stripped.



In past studies various formulations have been used to coat the interior walls of metal tubes. After drying and platinizing, the exact platinum laydown could only be determined by removing part of the coating and analyzing it for platinum content<sup>3)</sup>. Another approach has been studied, namely formulation in which one or more components has been preplatinized with an exactly known amount of platinum on powdered type 1 or type 16 support. These were then formulated with the usual type 6 binder and tested as 10-20 mesh granules. The adhesive properties and activities of 10-20 mesh granules are shown in Table 23. Generally, with a single platinized (either fibrous or particulate) type 1 support - type 6 binder adherence to smooth stainless steel is poor but adherence to sandblasted stainless steel (10860-163A and 161A) is fair. About the same results were obtained with a combination of fibrous and particulate type 1 supports (with binder) if either the fibrous support was preplatinized (10860-163C) or the particulate type 1 support was preplatinized (10860-163 and 161B). Somewhat better adhesion was obtained if both fibrous and particulate type 1 supports were preplatinized (10860-163E). Platinum contents varied from 1.6 to 3.2% overall in these catalysts, and the MCH dehydrogenation activity was as great as or greater than that of the reference catalyst. Similar experiments were conducted substituting preplatinized fibrous type 16 support for preplatinized type 1 support. The type 16 support is attractive as a possible substitute for the type 1 fibrous support no longer manufactured, a component in the ternary 40:40:20 type I formulation. For comparison, the results shown in Table 23 are obtained with a type 16 support - type 6 binder (80:20) which is first dried and then metallized with three concentrations of platinum (10860-160A, 160B, and 160C). Adhesion of the coating material is fair on smooth stainless steel and better on sand blasted stainless steel. The activity of the metallized dry granules is a little better than the reference catalyst. If the type 16 support is preplatinized (10860-167C) and then bonded, about the same adhesive properties to metal are obtained. Activity is a little less than for the 160 series of catalysts at the same platinum content. Premuffled fibrous type 16 support-preplatinized particulate type 1 support-type 6 binder (40:40:20, 10860-173) gives fair adherence to smooth stainless steel and good adherence to sand blasted stainless steel. The dehydrogenation activity is fairly high. If both supports are platinized and type 6 bonded, adherence is poorer to both smooth and sand blasted stainless steel (10860-175); activity of the platinized granules is about the same.

A recently available type 1 support with excellent physical properties has been studied as a coating candidate component and has shown excellent results in all respects, except for activity-stability on bench scale tests.

The data in Table 24 show that substitution of this new type 1 support into the usual 40:40:20 formulation (type I), in place of the most commonly used particulate type 1 support, gives good adhesion to aluminized and sand blasted stainless steel but not to smooth stainless steel surfaces (10860-176A). Rod milling improves the adhesion to smooth surfaces considerably (10860-178). Very high MCH dehydrogenation activities are obtained with the platinized granular counterparts (10860-177, 177B, and 184A). Equivalent results are obtained on rod milling the type I formulation containing the usual type 1 support component (10860-78B, 183, 81D, and 184E).

Table 24 follows

Quite divergent results are obtained on rod milling 80% newer type 1 support - 20% type 6 binder together (type II formulation). Adhesion is poor to both smooth and sand blasted stainless steel on mortar mixing but excellent after rod milling (10860-164 and 181). Catalyst activity is high and equivalent in both cases at the same platinum content (10860-164A-C, and 184C). With the previously used counterpart type 1 support included, adhesion is equally poor after either mortar mixing or rod milling with 20% type 6 binder. Again dehydrogenation activity is high at the same platinum content (10280-56A, 10860-182, 58A, and 184D). Thus, the use of the new type 1 support in the type I formulation appears to give a promising and simpler wall coating metal support than the previous corresponding support. However, another important factor, namely activity-stability, is yet to be determined.

Satisfactory adhesion to smooth, sandblasted and aluminized stainless steel surfaces has been obtained with fibrous type 16 instead of type 1 support formulations if mortar mixing is used, but poor adhesion to a smooth surface results with ball milling (176B and 180). A small penalty in activity is taken in both cases (10860-177C, 177D and 184B). The activity-stability of this type of catalyst has not been studied as yet in bench scale studies.

#### Aluminum Clad Stainless Steel

Sources of internally aluminum clad stainless steel or Hastelloy C tubes are being sought. The oxidized aluminum surface is expected to form an even better bonding surface than the bare stainless steel walls for coating formulations, and may itself be a suitable support for impregnation with very thin coatings of metals catalytically active for dehydrogenation reactions. Coated tubes of these types should have better resistance to thermal strains set up by differences in thermal expansion between metals and catalytic coating materials.

One type of aluminum diffusion alloyed 1/4" OD stainless tubing was obtained from Alon Processing Company. These were unsatisfactory in that about one-half of the tubes were plugged solid, and all tubes had rough, uneven, and very rusty internal surfaces. The exteriors had a very rusty appearance and copiously shed rust flakes. The ends of tubes were magnetic, more so than the centers, apparently because magnetic austenite was formed at high temperatures in the cladding treatment. Emission spectroscopy verified that the material was 304 type stainless steel, and this was later confirmed via telephonic conversation with a representative of Alon Processing Company. The poor condition of the tubes did not warrant catalytic coating studies with them.

Four stainless steel strips which had been aluminized on one side were obtained from the above company. These strips had the usual general grey metallic appearance of stainless steel and were free from rust. The aluminized sides had a dull grey, rough, pebblegrain appearance. On heating in air at 1292°F a slight white surface oxidation was observable. Coating material 10860-124, after drying and muffling, adhered well to the aluminized but unoxidized surface of a small strip. The coating principally filled the indentations between the pebblegrains. In view of the unsatisfactory physical condition of the above tubes, no further experimentation with coating them catalytically is contemplated.

Table 24. MICR EVALUATION AND METAL ADHESIVE PROPERTIES OF VARIOUS PLATINIZED  
WALL COATING CANDIDATES

Test Conditions: Feed Stock MCH, LHSV 100, 10 atm, no added hydrogen,  
(0.9 ml 10-20 mesh catalyst diluted to 2.0 ml  
volume with quartz).  
GLC samples taken at 3, 8, and 13 minutes, respectively.

Support			Metal Adhesive Properties					Catalyst 10860-	% Pt	MICR Run No.	Conv. of MCH To Toluene, 752°F
10860-	Composition	Stainless Steel		Aluminized SS							
		Thickness	% Stripped <sup>a)</sup>	Thickness	% Stripped <sup>a)</sup>						
--	JOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	--	--	--	--	--	70	1	1232	46	
164	80% type 1 support - 20% type 6 binder (not rod milled)	7 mils (SM) 9 mils (SB)	100 100	--	--	--	164A 164B 164C	1 2 4	1215 1216 1217	49 55 57	
181	80% type 1 support - 20% type 6 binder (rod milled)	7 mils (SM) 6-7 mils (SB)	20 5	3-4	5-10		184C	4	1250	59	
176A	40% type 1 support (1) - 40% type 1 support (2) - 20% type 6 binder (not rod milled)	2 mils (SM) 5 mils (SB)	95 5	6-7	5		177A 177B	3 4	1244 1245	54 55	
176	40% type 1 support (1) - 40% type 1 support (2) - 20% type 6 binder (rod milled)	2-3 mils (SM) 2-3 mils (SB)	5 0	2-3	0		184A	4	1248	55	
10860- 5CA	80% type 1 support - 20% type 6 binder (not rod milled)	-- (SM) -- (SB)	-- 100	--	--		10280- 58A	4	308	~55	
182	80% type 1 support - 20% type 6 binder (rod milled)	3-4 mils (SM) -- (SB)	90 90	3-4	70		184D	4	1251	54	
783	40% type 1 support (1) - 40% type 1 support (2) - 20% type 6 binder (not rod milled)	-- (SM) -- (SB)	100 <sup>b)</sup> 20	--	--		81D	2	965	57	
183	40% type 1 support (1) - 40% type 1 support (2) - 20% type 6 binder (rod milled)	3-4 mils (SM) 1-2 mils (SB)	5 0	3-4	0		184E	4	1252	53	
176B	40% type 16 support - 40% type 1 support - 20% type 6 binder (not rod milled)	8 mils (SM) 7 mils (SB)	5 0	5	20		177C 177D	3 4	1246 1247	51 51	
180	40% type 16 support - 40% type 1 support - 20% type 6 binder (rod milled)	4 mils (SM) 4 mils (SB)	65 20	2	10		184B	4	1253	50	

SM = Smooth degreased stainless steel.

SB = Sand blasted stainless steel.

SS = Stainless Steel.

a) % Stripped off when pressure sensitive tape pressed onto surface, and then gently pulled off.  
b) Self stripped during drying.

Various stainless steel and Hastelloy C tubes of different lengths and diameters and several strips of stainless steel have been obtained that are aluminum clad on all exposed surfaces. These have been clad by a proprietary vacuum diffusion process (PNA-73) by Pratt and Whitney Aircraft Division. Preliminary experiments have been carried out by coating one side of the aluminized strips with the more recent experimental coating formulations. These tests have been carried out at the same time as those with the smooth and sand blasted stainless steel strips, and the data are included in Tables 23 and 24. Generally, coating adherence is about equivalent to that of sand blasted stainless steel and superior to that of smooth stainless steel. Preoxidation of the aluminized surface in air at 1382°F appears to increase slightly the tenacity of the coating adhesion.

#### MEASUREMENT OF CATALYTIC REACTION KINETICS

Quantitative data on the chemical kinetics of MCH dehydrogenation have been obtained previously in a tubular flow reactor with packed bed catalyst. A single stoichiometric reaction is involved, constituting fairly simple kinetics. Even so, difficulties were encountered with temperature and concentration gradients in the packed bed and in the catalyst pellets. The gradients in the packed bed were accounted for by calculating them with a rather involved computer program, and also by attempting to minimize these gradients by changing the reactor geometry. The gradients in the catalyst pellets were not fully taken into account and have only been fully appreciated recently.<sup>3)</sup> The result of these interfering nonkinetic effects of heat and mass transfer is that the true chemical kinetics are difficult to extract from the experimental data.

#### Need For Intrinsic Kinetic Data

A legitimate question that could be raised is: Why is it necessary to determine the true chemical kinetics? Since in the full scale reactor heat transfer and mass transfer effects will be present, why not just run the experiments at the desired conditions and fit a model to the results, regardless of whether the effects are due to mass transfer, heat transfer, or the intrinsic chemical kinetics? The answer stated simply is that is is extremely difficult (often impossible) to exactly simulate the conditions of the final application. Furthermore, if one were to obtain such a model based on this completely empirical approach, extrapolation of the model to conditions other than those of the experiments would be highly questionable. As a straightforward example, consider an experiment performed to measure the rate of chemical reaction using a catalyst pellet 1/4" in diameter. One could certainly measure this quantity and fit a chemical kinetic model to it. The problem would arise if the measured rate of reaction was affected by the rate of diffusion of reactants and products through the pores of the catalyst. An attempt to use the kinetic expression for the same catalyst as a 1/8" diameter pellet would fail, since the diffusion would occur much more rapidly than before, resulting in a higher net rate of reaction. The kinetic model would not predict this since the pellet size and shape is not part of the kinetic expression. A similar problem could occur by changing the velocity of the fluid flowing past the catalyst. This can change the rate of heat and mass transfer to the catalyst pellet and thereby the apparent rate of reaction, whereas a kinetic model would not account for this and would miss the effect entirely. A broad experimental

study could be made of the effect of all of the important variables in the reactor such as reactor configuration, pellet shape and size, catalyst composition and structure, and fluid pressure, temperature, and velocity. But one rapidly finds that an enormous amount of experimental work would be required, and the resulting model would simply be a device for representing the data.

A more satisfactory procedure is to separate the different processes which occur, study each independently, and then recombine them to form a model which often has a sound fundamental basis and can be used fairly confidently to calculate results, even in regions where experimental data are not available. In heterogeneous catalysis, there are several processes which can often be separated. Much is known about the rate of heat and mass transfer to and from inert particles (e.g., spheres). This information can be applied to a model of the processes surrounding a spherical catalyst pellet. Models are also available for the rate of diffusion in the pores of the catalyst and the rate of heat transfer within the catalyst pellet. Also, much information is available on the mechanisms of catalytic chemical reactions. Assuming one can obtain kinetic information in the absence of the interfering effects of heat and mass transfer, mechanistic models can be proposed, tested, evaluated, and fitted. Once a good mechanistic model is found which relates the rate of reaction to concentrations, pressures, and temperatures, then the models of heat and mass transfer can be applied to result in a reasonable mathematical model with the desired characteristics.

#### Design of Experimental Reactors

The problems in designing a laboratory reactor for catalytic chemical kinetics has been widely discussed in the literature.<sup>6,7)</sup> The problems are exactly those described above. A system is required in which the interfering effects of heat and mass transfer are either absent or mathematically describable. The reactor type used in the traditional approach, and the one used in this investigation in the past, has been a packed tubular reactor. The problems with this reactor are that the flow patterns are complicated, and there are invariably appreciable temperature and concentration gradients between catalyst particles as well as within particles. Also, the reactor produces integral rate data and not the rate at a particular set of conditions. Differential reactors are sometimes used to eliminate the last problem, but errors in the chemical analysis lead to large errors in rates. To remedy these problems two different kinds of reactors have been developed: the recycle reactor and the catalytic continuous stirred tank reactor. In both the conditions of reaction are uniform and well defined, and many of the heat and mass transfer problems can be eliminated. In both reactors the reaction rate is measured directly from the conversion and the flow rates.

A recycle reactor consists of a small amount of catalyst exposed to a flowing stream of reactant, most of which is being recycled past the catalyst. During each cycle only a small amount of conversion is occurring, but the total conversion of feed to product can be as high as desired for easy analysis. Such reactors have been used by Butt et al.<sup>8)</sup> and Perkins and Rase<sup>9)</sup>. About the only problem with this kind of reactor is the pump used to recycle the fluid. It must meet rigid standards of noncontamination and be able to withstand the operating conditions. Bernard and Teichner<sup>10)</sup> describe a novel pump.

A catalytic continuous stirred tank reactor (CCSTR) has essentially the same advantages as the recycle reactor. In this type the catalyst is either contained in the impeller or is mounted on the wall, and the reactant enters, becomes well mixed, reacts, and leaves. Again, virtually any conversion is feasible. Reactors of this type have been used by several workers.<sup>11-13</sup> The problem here is one of agitation. Sufficient agitation must be supplied to the fluid phase to ensure complete mixing. In a high temperature, high pressure system special equipment is required. Since this type of reactor has been found to work well at high temperatures and pressures, we have chosen it for our work on the dehydrogenation kinetics of the MCH and decalin systems.

The catalytic continuous stirred tank reactor which we have designed will be a modification of one of our existing standard reactors made by Autoclave Engineers. The agitation will be supplied by a magnetic drive with speeds up to about 2000 rpm. The reactor is designed to operate to 1000 psig at 1000°F. It will be constructed of Inconel and be about 1 liter in size. The impeller will be constructed of parallel screens containing catalyst pellets.

#### MATHEMATICAL MODEL OF CATALYTIC WALL REACTOR

A mathematical model has been developed for our catalytic wall reactor (CWR). The model should be useful in analyzing our experiments and in estimating the effects of changes in operating conditions, reactor geometry, and catalyst composition. The model assumes that the fuel flows turbulently through a tube which has a thin coating of catalyst on the wall. The fuel is assumed to be well mixed radially so that the bulk flow may be considered to be one-dimensional. The heat is assumed to be applied by generation in the tube wall, since that is how it is done in the experiments with the Fuel System Simulation Test Rig (FSSTR). The heat transfer between the outside of the tube and the environment is given as a boundary condition. The model then accounts for the heat transferred radially between the tube wall and the catalyst layer, and between the catalyst layer and the bulk fluid flow. It also calculates the axial temperature, pressure, and conversion profiles.

#### Model Development

The equations for the bulk gas and for the tube wall were adapted from those derived previously for the regenerative heat exchanger model.<sup>2)</sup> The equations describing the bulk fluid flow result from a one-dimensional momentum balance, an energy balance, and a mass balance (see Appendix):

$$\frac{d}{dz} \left( \frac{\partial p}{\partial z} \right) + \frac{d}{dz} \left( \frac{\partial T}{\partial z} \right) - \frac{\rho d}{G^2} \left( \frac{dP}{dz} \right) - 2f = 0 \quad (1)$$

$$\frac{\rho^2 c_p d}{G^2} \left( \frac{dT}{dz} \right) + \frac{T d}{G^2} \left( \frac{dP}{dz} \right) + \frac{4\rho^2 \alpha l}{G^3} - 2f = 0 \quad (2)$$

where

$d$  = inside catalyst diameter  
 $\rho$  = fluid density  
 $P$  = total pressure  
 $Z$  = axial distance  
 $T$  = temperature of bulk fluid  
 $G$  = mass flux of fluid  
 $f$  = Fanning friction factor  
 $c_p$  = fluid specific heat at constant pressure  
 $q_1$  = heat flux from fluid to catalyst layer (based on inside diameter of catalyst)

Equations (1) and (2) may be solved for the derivatives of  $P$  and  $T$  with respect to  $Z$ :

$$\frac{dP}{dZ} = \frac{G^2}{\rho d} \left( \frac{a_{22}b_1 - a_{12}b_2}{a_{11}a_{22} - a_{12}a_{21}} \right) \quad (3)$$

$$\frac{dT}{dZ} = \frac{G^2}{\rho^2 d} \left( \frac{-a_{21}b_1 + a_{11}b_2}{a_{11}a_{22} - a_{12}a_{21}} \right) \quad (4)$$

where

$$a_{11} = -1 + \frac{G^2}{\rho^2} \frac{\partial \rho}{\partial P}$$

$$a_{12} = \frac{G^2}{\rho^3} \frac{\partial \rho}{\partial T}$$

$$a_{21} = \frac{T}{\rho} \frac{\partial \rho}{\partial T}$$

$$a_{22} = c_p$$

$$b_1 = 2f$$

$$b_2 = 2f - \frac{4\rho^2 q_1}{G^3}$$

Equations (3) and (4) are integrated numerically to give axial temperature and pressure profiles of the bulk fluid flow. The following data are required to solve these equations:

1. The density at each axial position.
2. The partial derivatives of the fluid density with respect to pressure and temperature.

3. The heat capacity at constant pressure.
4. The Fanning friction factor.
5. The rate of heat transfer to and from the catalyst layer.
6. The composition of fluid at each axial position.

The required physical properties are calculated by techniques discussed previously.<sup>3)</sup> In addition to density, density derivatives, and heat capacity, the viscosity and thermal conductivity must be calculated. Suitable subroutines were written to do this. The Fanning friction factor is calculated by the equation presented by Koo<sup>17)</sup>, modified slightly:

$$f = 0.0014 + \frac{0.125}{(\text{Re}')^{0.32}} \left( \frac{T}{T_i} \right)^{0.5} \quad (5)$$

where

Re' = modified Reynolds number =  $(dG/\mu) (T/T_i)$   
 $\mu$  = viscosity at wall  
 T = bulk absolute temperature  
 T<sub>i</sub> = wall absolute temperature

The rate of heat transfer is based on film theory using the Dittus-Boelter correlation for the heat transfer coefficient:

$$h = 0.023 \frac{k}{d} (\text{Re})^{0.8} (\text{Pr})^{0.4} \quad (6)$$

where

h = heat transfer coefficient  
 k = thermal conductivity of film  
 Pr = Prandtl number of film  
 Re = Reynolds number of film

The differential equation describing the conversion is derived in the Appendix. The equation is

$$\frac{dx}{dZ} = \frac{r}{m} v_c \quad (7)$$



where

x = number of moles reactant converted per unit feed  
 r = rate of reaction per unit volume of catalyst (kinetic expression)  
 ṁ = total mass flow rate  
 v<sub>c</sub> = volume of catalyst per unit length

The temperatures in the catalyst layer and in the tube wall are calculated assuming that the rate of heat generation or absorption is uniform across each section. With this assumption analytical expressions may be derived (see Appendix) for the temperature rise across each section, and the average temperatures can be estimated. The temperature rise across the catalyst layer is found from

$$\Delta T_{cat} = \frac{q_R' y_2}{2k_c} \left[ \frac{1 - \left(\frac{y_2}{y_1}\right)^2 + 2 \ln \left(\frac{y_2}{y_1}\right)}{1 - \left(\frac{y_2}{y_1}\right)^2} \right] - \frac{q_1' y_2}{k_c} \ln \left(\frac{y_2}{y_1}\right) \quad (8)$$

And the temperature rise across the tube wall is

$$\Delta T_{wall} = - \frac{q_G' y_2}{2k} \left[ \frac{1 - \left(\frac{y_2}{y_3}\right)^2 + 2 \ln \left(\frac{y_2}{y_3}\right)}{1 - \left(\frac{y_2}{y_3}\right)^2} \right] + \frac{q_3' y_2}{k} \ln \left(\frac{y_2}{y_3}\right) \quad (9)$$

where

$\Delta T_{cat}$  = temperature increase from inside to outside of catalyst layer  
 $q_R'$  = heat flux to chemical reaction, based on inside tube area  
 $y_2$  = inside radius of tube  
 $k_c$  = thermal conductivity of catalyst  
 $y_1$  = inside radius of catalyst layer  
 $q_1'$  = heat flux from fluid to catalyst, based on inside tube area  
 $\Delta T_{wall}$  = temperature increase from inside to outside of tube  
 $q_G'$  = heat flux of heat generated in tube wall, based on inside tube area  
 $k$  = thermal conductivity of tube wall  
 $y_3$  = outside radius of tube  
 $q_3'$  = heat flux from outside of tube to ambient conditions, based on inside tube area

The average temperatures in the catalyst and the wall are estimated by assuming that the temperature differences can be divided into a generation part (terms involving  $q_g^i$  and  $q_g^j$ ) and a transfer part (terms containing  $q_t^i$  and  $q_t^j$ ). Heat generation leads to approximately parabolic temperature profiles, while heat transferred leads approximately to linear profiles. The average temperatures then are found by using  $1/3$  of the difference due to generation and  $1/2$  of the difference due to transfer.

At the present time, the model assumes that there is no diffusional resistance between the bulk fluid and the interior of the catalyst. In the language of catalysis, this assumes external and internal effectiveness factors of one, and can be approached by high fluid velocities and thin catalyst layers.

The calculation of the radial heat fluxes and temperature profiles involves a trial and error procedure as follows:

1. The average catalyst temperature is assumed.
2. The rate of reaction is then calculated from the kinetic expression.
3. The heat absorbed by the reaction can then be calculated.
4. The heat flux from the tube wall to the catalyst is found as the difference between the heat generated in the tube wall and the heat lost from the outside of the tube.
5. The difference between the heat flux from the tube wall to the catalyst and the heat absorbed by the reaction is the heat flux between the catalyst and the fluid.
6. Knowing the temperature of the fluid and the heat flux to or from the fluid, the temperature of the inside catalyst surface can be found using the Dittus-Boelter equation.
7. Assuming that the heat absorbed by the reaction is constant radially, the radial temperature profile may be calculated by equation (8).
8. Then an improved value of the average reaction temperature can be found by a simple convergence procedure, and the process is repeated.

The key assumptions in this calculation are that the average reaction rate takes place at the average temperature and that the heat uptake by the reaction is uniform radially. Neither assumption is strictly true. The reaction rate is an exponential function of the temperature so that the average temperature will underestimate the average rate. The other assumption, however, tends to counteract the error. Since the reaction is endothermic, the hotter part of the catalyst, where the reaction occurs faster, is cooled more by the reaction, thereby retarding the rate.

### Application to Methylcyclohexane Dehydrogenation

Only two experiments have been performed with catalytic wall reactors under realistic flow conditions. The runs were made in the FSSTR and were reported earlier.<sup>3)</sup> Test series 10018-167 was chosen for comparison with the model. The experiments were conducted at three power input levels: 0, 95,000, and 195,000 Btu/hr-ft<sup>2</sup>, all at 860 psig pressure and 930°F fluid inlet temperature. The feed rate was 19.7 lb MCH per hour, which gave a Reynolds number of about 100,000 in the highly turbulent flow regime.

The computer program required the heat losses to ambient conditions, which were obtained from the measured outside wall temperatures during the run and a knowledge of heat losses as a function of temperature, which had been obtained experimentally under no flow conditions. The critical constants of methylcyclohexane and toluene were supplied along with a set of pseudocritical constants for hydrogen:

$$T_c = 43.6^\circ\text{K}$$

$$P_c = 20.2 \text{ atm}$$

$$V_c = 51.5 \text{ cm}^3/\text{gmole}$$

These constants for hydrogen yield more accurate results than the true ones for corresponding states correlations. The thermal conductivity of the catalyst was estimated to be 0.15 Btu/hr-ft-°F, based on data of Sehr<sup>18)</sup> on similar catalysts. The thermal conductivity of the tube wall was calculated by

$$k = 7.33 + 0.00458 T_w \quad (10)$$

where

$$T_w = \text{temperature of the tube wall, } ^\circ\text{F}$$

The kinetic expression used was that obtained from packed bed experiments:<sup>2)</sup>

$$r = \frac{k_1 c}{1 + k_2 c} \left( 1 - \frac{p_T p_H^3}{p_M K_e} \right) \quad (11)$$

where

$r$  = rate of reaction of MCH per unit volume of catalyst, lb mole/hr-ft<sup>3</sup>

$c$  = concentration of MCH, lb mole/ft<sup>3</sup>

$k_1 = A_1 \exp(B_1/RT)$

$k_2 = A_2 \exp(B_2/RT)$

$p_T$  = partial pressure of toluene, atm

$p_H$  = partial pressure of hydrogen, atm

$K_e = A_3 \exp(B_3/RT)$

$R$  = universal gas constant = 1.987 Btu/lb mole  $^{\circ}R$   
 $T$  = absolute temperature,  $^{\circ}R$   
 $A_1 = 5.4 \times 10^5 \text{ hr}^{-1}$   
 $B_1 = -5000 \text{ Btu/lb mole}$   
 $A_2 = 4.5 \times 10^{-8} \text{ ft}^3/\text{lb mole}$   
 $B_2 = 54,000 \text{ Btu/lb mole}$   
 $A_3 = 4.0 \times 10^{20} \text{ atm}^2$   
 $B_3 = -92,500 \text{ Btu/lb mole}$

It was found that 50 integration steps were sufficient to obtain an accurate solution to the equations.

The results using the packed bed kinetic parameters are summarized in Figure 10. The conversion of MCH at the three different power levels is shown, as is the outlet fluid temperatures. The experimental values are represented by the discrete points, and the calculations by solid lines. As can be seen, the conversions are calculated much lower than those found experimentally. The fluid temperatures are therefore much higher, since the transferred heat not absorbed by reaction must heat the fluid. The fact that the conversions are calculated too low indicates that either the catalyst temperatures are calculated too low or that the kinetic expression predicts a rate which is too low. The first possibility can be eliminated since the wall temperatures are calculated much higher than those observed experimentally, and as a result the catalyst temperatures are actually predicted too high. Therefore the packed bed kinetic parameters predict too low a rate of reaction. This is probably due to pore diffusional limitations, which are calculated to be appreciable in the catalyst pellets used in the packed bed. The thin layer of catalyst used in the catalytic wall experiments (3 mils) would not show the same diffusional limitations.

Several variations in the parameters  $A_1$ ,  $B_1$ ,  $A_2$ , and  $B_2$  were made to determine their effect on the results. The most reasonable results with the least effort were obtained by increasing the value of  $A_1$  only. A value of  $30. \times 10^5$ , rather than  $5.4 \times 10^5$ , gave the best agreement with the data. The results are shown in Figure 11. The conversions at the two higher power levels are calculated slightly too high. The outlet fluid temperatures are also too high, indicating a small error in the heat balance. The fluid temperature at zero power level is  $40^{\circ}F$  high, which is somewhat greater than the others. In general, though the results are encouraging. The lack of heat balance is easily explained by a loss of heat between the points at which the fluid temperatures are measured and the beginning and end of the reactor, which is not included in the calculations. If  $910^{\circ}F$  is used as the entering temperature, instead of  $930^{\circ}F$ , the heat balance checks out. Figure 12 shows the result for using  $910^{\circ}F$  initial fluid temperature. The calculated temperatures and conversions agree quite well with the experiments at the two higher power levels, and are not very far off at zero power.

A comparison between the calculated and experimental outer tube temperatures is shown in Figure 13. The agreement is not excellent, but except for the first few inches the results agree within  $40^{\circ}F$ . It became apparent during the experimental run that something was wrong with the first 4 inches of catalyst. The outer wall temperatures climbed steadily with time indicating that the catalyst was deactivating. This could be explained in terms of coke formation on the

catalyst; however, the phenomenon affected only the first four inches and apparently did not progress downstream. It was not clear why the apparent lack of activity only occurred in the initial region of the reactor. Part of the answer may lie in an indication that the catalyst was thicker in this region. Outer temperature profiles were obtained during the activation of the catalyst with hot nitrogen gas. The profiles for no input power and a low power level are shown in Figure 14. At no power input the initial temperatures appear to be too low. When power was applied, the temperatures appeared to be too high. This seems to indicate that the catalyst was thicker in the first four inches of length, which would cause at least part of the greater temperature differences observed. However, this apparent lack of activity may also be due to lower partial pressure of hydrogen or poisoning of the catalyst at the inlet end of the reactor. Further experiments are clearly in order to resolve this question.

The fluid pressure was measured to be 860 psig at the end of the reactor for all three power levels. This pressure was calculated by the model to be 853, 851, and 848 psig in order of increasing power input.

#### Accuracy of Predicted Reaction Rates

In an effort to check the effect of the several assumptions concerning the temperature profile and rate of reaction in the catalyst layer, a detailed calculation of the radial variations was carried out. A set of conditions at the 195,000 Btu/hr-ft<sup>2</sup> heat flux during experimental run 10018-167 was used. The CWR model gave an average reaction rate of 10,600 lbmole/hr-ft<sup>3</sup> cat. and an average reaction temperature of 911°F. The inner and outer surfaces of the catalyst were calculated to be 892 and 1006°F, respectively. These conditions were determined for a position 0.037 ft from the entrance of the reactor.

The differential equation for the radial temperature profile of the catalyst is

$$\frac{d^2T}{dy^2} + \frac{1}{y} \frac{dT}{dy} = \frac{r\Delta H_R}{k_c} \quad (12)$$

where

- T = temperature of catalyst, °F
- y = radial distance, ft
- r = reaction rate, lbmole/hr-ft<sup>3</sup>
- $\Delta H_R$  = heat of reaction, Btu/lbmole
- $k_c$  = thermal conductivity of catalyst, Btu/hr-ft<sup>2</sup>°F

This equation was integrated numerically using the inner catalyst surface temperature of 892°F and the known heat flux at the surface between the catalyst and the tube wall. The temperature, the reaction rate, and the concentration were allowed to vary during the integration. The results for the temperature profiles are

Figures 10 through 14 follow

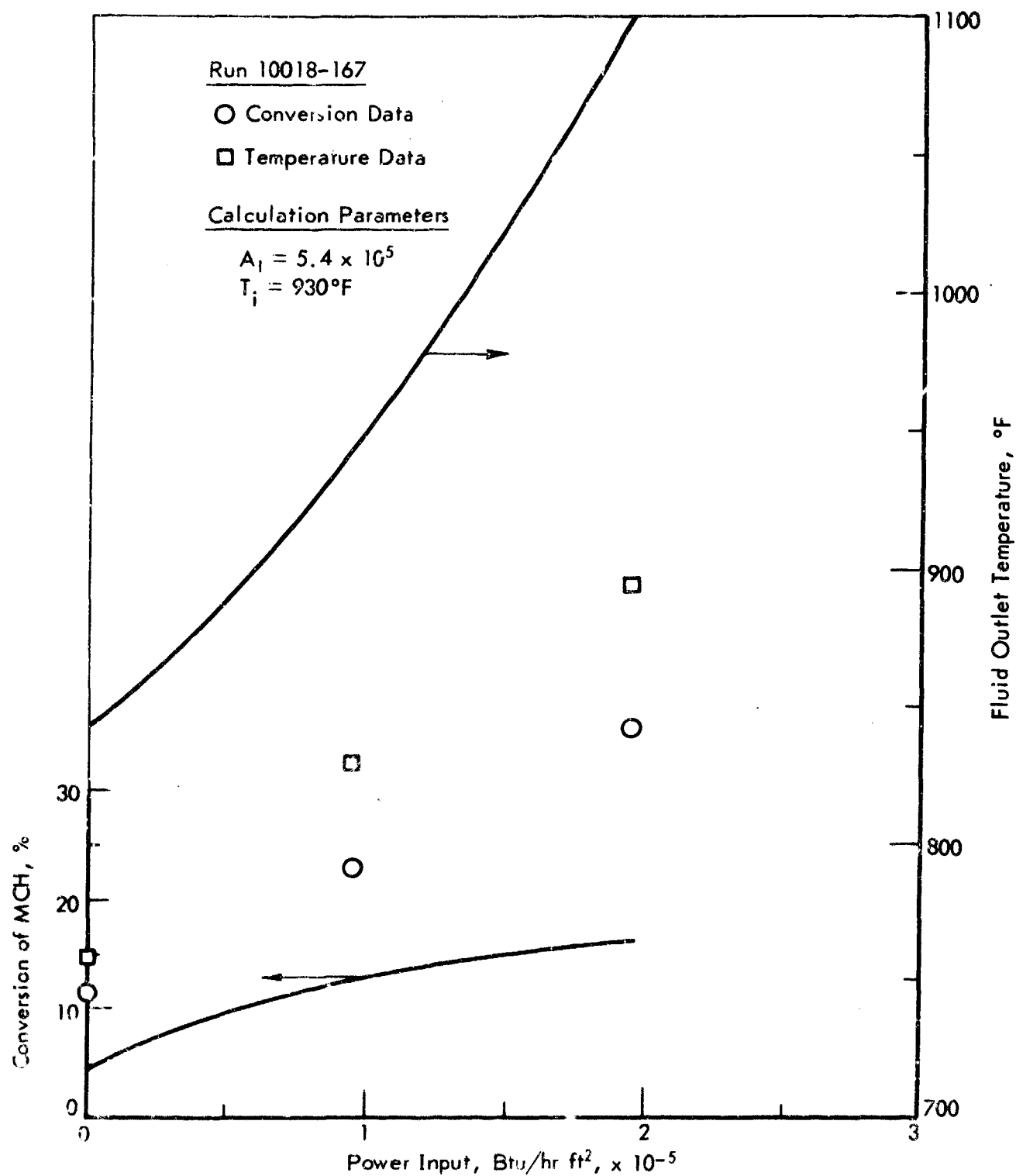


Figure 10. METHYLCYCLOHEXANE DEHYDROGENATION IN A WALL CATALYST

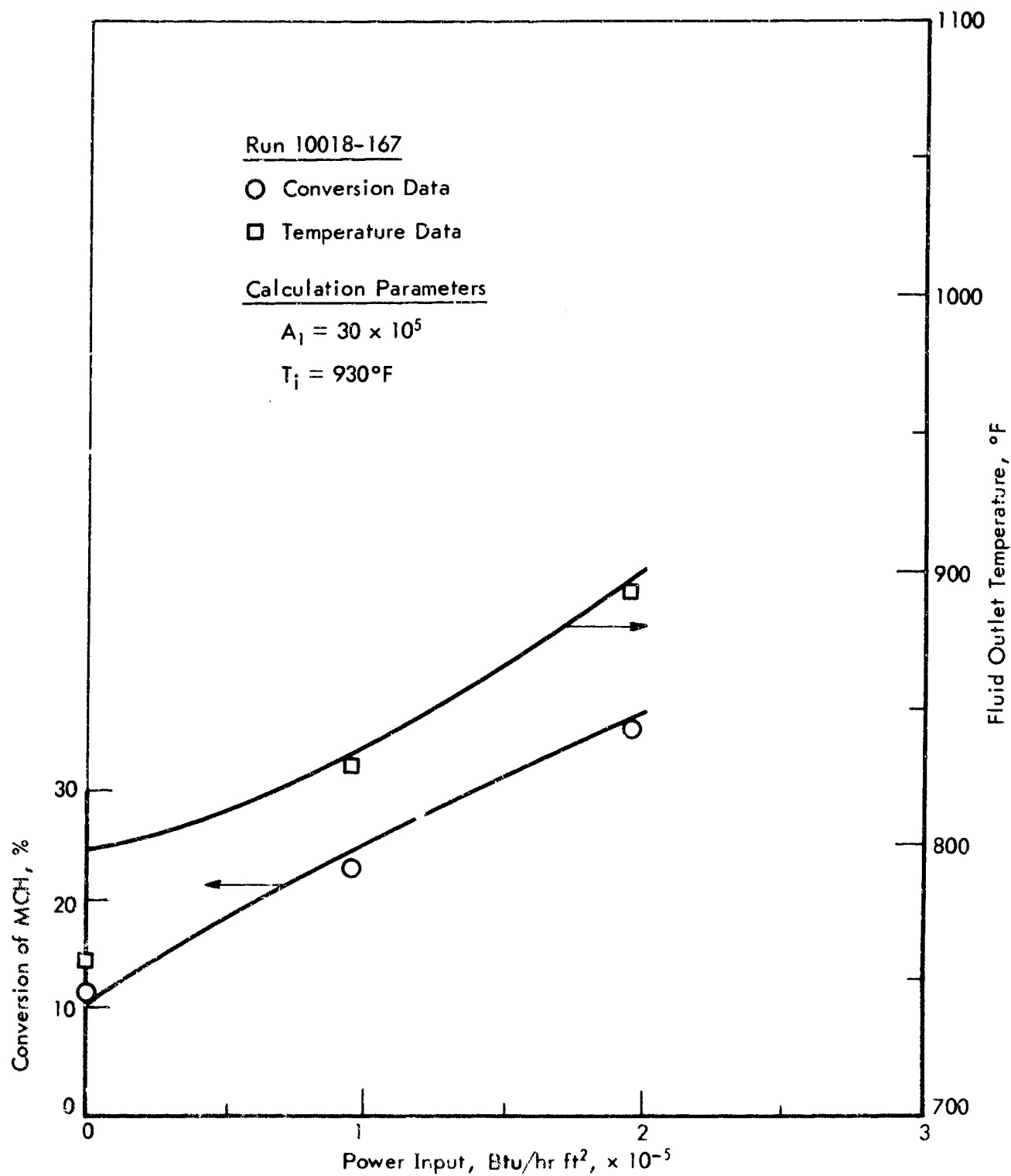


Figure 11. METHYLCYCLOHEXANE DEHYDROGENATION IN A WALL CATALYST

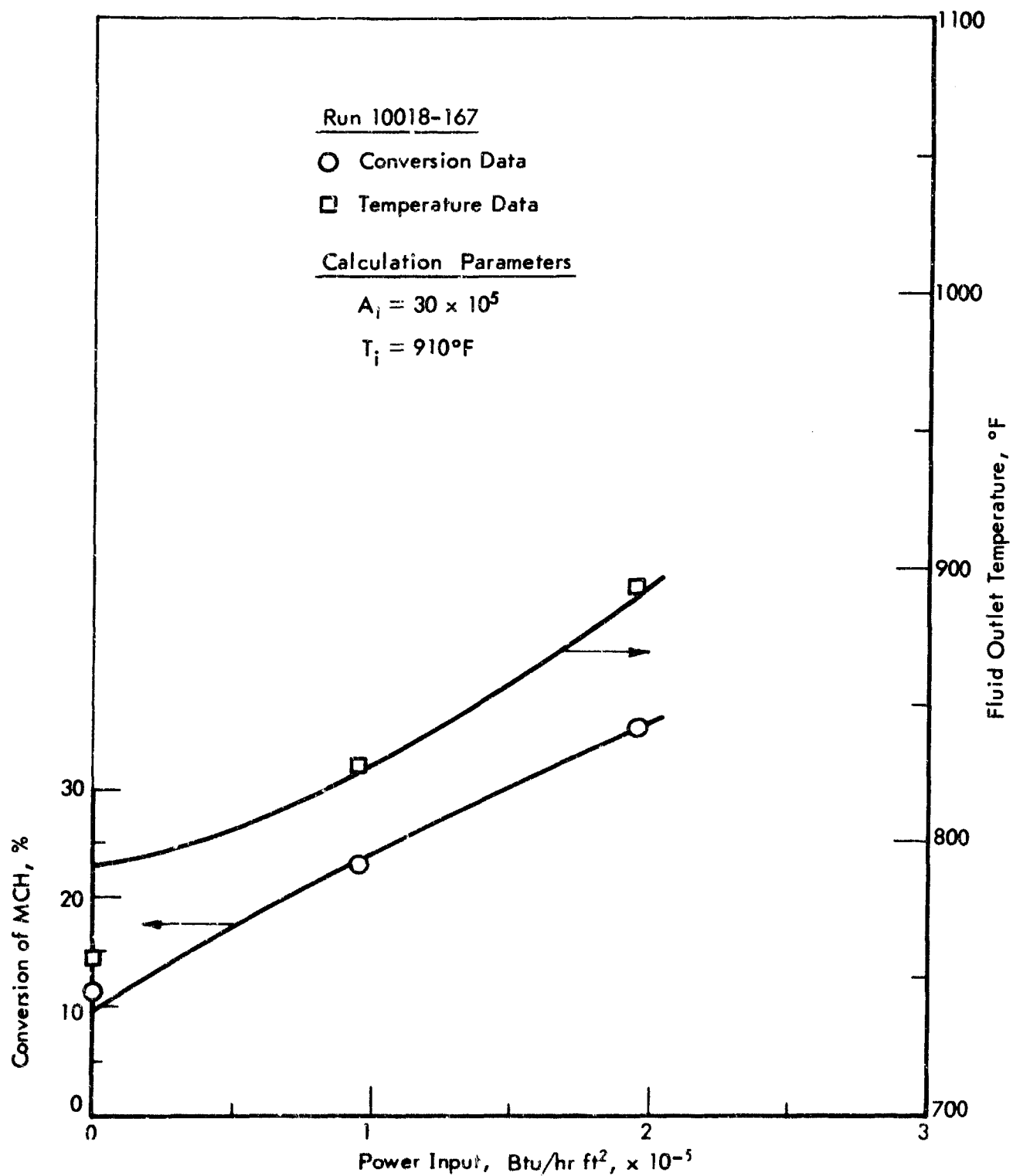


Figure 12. METHYLCYCLOHEXANE DEHYDROGENATION IN A WALL CATALYST



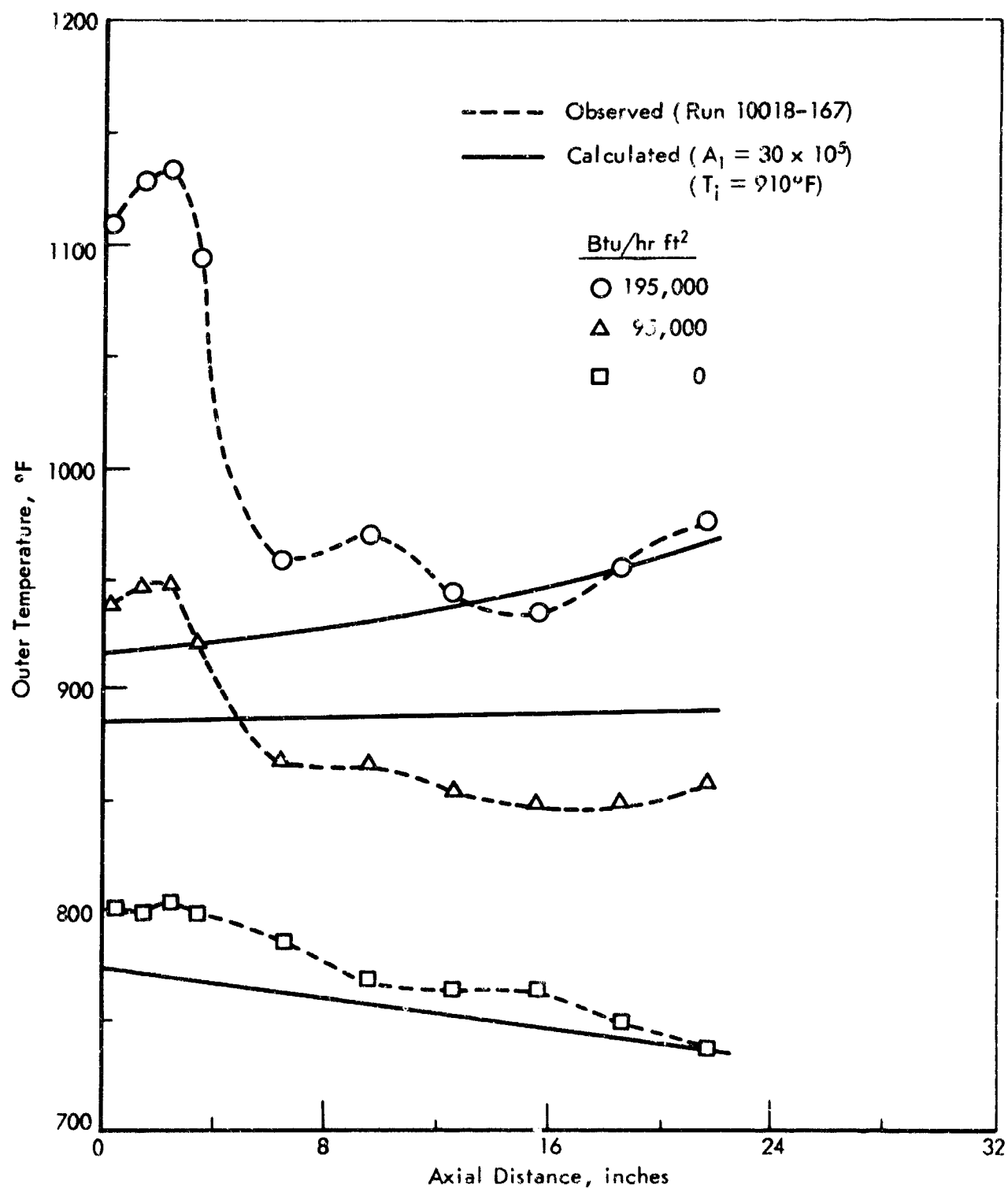


Figure 13. OUTER WALL TEMPERATURES DURING METHYLCYCLOHEXANE DEHYDROGENATION IN A WALL CATALYST

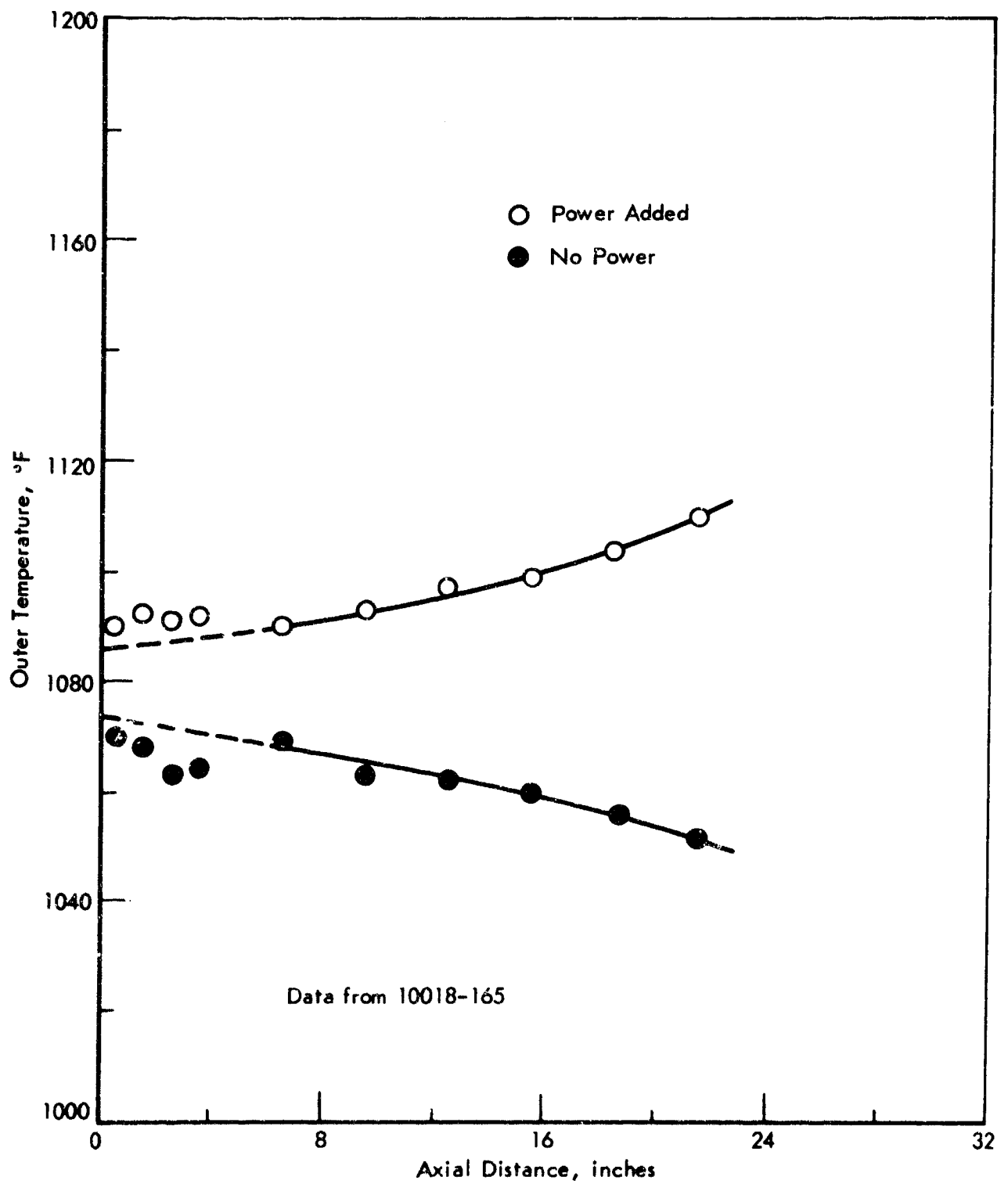


Figure 14. OUTER WALL TEMPERATURES DURING ACTIVATION OF WALL CATALYST

shown in Figure 15. As was expected the temperatures resulting from the radially integrated equation gave a flatter profile (solid curve), reflecting the additional cooling in the region of highest temperature. The profile resulting from the analytical solution in the CWR model, assuming uniform heat sink (dashed line), gave more temperature variation and a higher outer surface temperature.

The average temperature of the catalyst calculated by the radial integration is 900°F, and the reaction rate is 9300 lbmole/hr-ft<sup>3</sup>. This corresponds to an error in the CWR model calculation of about 1.2% in the temperature and about 14% in the reaction rate and conversion. The results were obtained by comparison with the experiments at the most extreme conditions we have run. At lower heat fluxes the errors should be less. One solution to this problem would be to integrate radially as well as axially; however, the execution times probably would be some excessive. Instead, there may be a way to obtain a better estimate of the average reaction temperature without resorting to this two-dimensional approach.

#### DEHYDROGENATION OF DECALIN IN THE FUEL SYSTEM SIMULATION TEST RIG

The reactor (3/8" OD x 0.049" wall x 24" long Hastelloy C) used in these tests has been used previously in this program. However, during its last use a leak developed in one of the welds and this had to be repaired. Since several of the attached thermocouples were also damaged, the ceramic coating was completely removed from the tube and all couples were replaced. A sketch of the reactor is given in Figure 16.

The following three test series have been completed using the indicated catalysts charged to the reactor section in the Fuel System Simulation Test Rig (FSSTR).

Series 10018-194	UOP-R8 catalyst (ca 0.8% Pt/ 1/16" Al <sub>2</sub> O <sub>3</sub> spheres)
Series 10018-198	Shell 10280-113 catalyst (ca 4% Pt/ 1/16" Al <sub>2</sub> O <sub>3</sub> spheres)
Series 11644-6	Shell 10860-146 catalyst (ca 4% Pt/10-20 mesh Al <sub>2</sub> O <sub>3</sub> )

The normal operating sequence for these tests was as follows:

- 1) Charge catalyst to reactor and activate by heating in nitrogen for 1 hr at 1100°F.
- 2) Set feed rate at 25.0 lb/hr (LHSV = 545).
- 3) Set inlet pressure at 900 psig.
- 4) Establish inlet temperature at 900°F by preheating feed in two 3/8" OD x 10' long heat exchange sections.

Figures 15 and 16 follow

- 5) Operate long enough with no power supplied to the reactor to establish steady state conditions. (Outlet fluid temperature was ca 720°F under these conditions.)
- 6) Supply sufficient power to reactor to raise outlet temperature to 800°F. Maintain power constant for ca 1 hr.
- 7) Increase power to reach 900°F outlet and run for ca 1 hr at constant power.
- 8) Increase power to reach 1000°F outlet and run at constant power long enough to establish catalyst activity decline rate.
- 9) Decrease power to bring outlet temperature down to 800°F again and hold long enough to establish final catalyst activity.

When the outlet temperature is changed to a new value in this operating procedure, the initial power requirement is noted and maintained constant for that particular test period. By following this procedure it is possible to tell when the catalyst activity is decreasing, as a decrease in conversion at constant power input results in an immediate increase in fluid outlet temperature. Of course, product analyses also show the decline in activity, but these are not available until well after a run has been completed.

The three catalysts exhibited quite different characteristics. The UOP-R8 and Shell 113 catalysts had similar initial activity up to 900°F outlet fluid temperature, but the Shell 113 catalyst declined in activity so rapidly at this power input that the outlet temperature had increased to 1000°F at the end of one hour. Hence, no further power increase was made. The Shell 146 catalyst not only had much higher initial activity than either of the other two catalysts but also exhibited superior stability. Figure 17 shows the initial conversion attained by the three catalysts at outlet fluid temperatures up to 900°F before any deactivation had occurred. Table 25 summarizes the conversion and outlet temperature history for the three test runs. Decline in conversion and the accompanying rise in outlet fluid temperature are evident for the three catalysts at power levels sufficient to give 900°F or higher outlet temperatures.

Operating data and product analyses are tabulated and fluid temperatures and decalin conversions are plotted against run time for each of the tests in the following Tables and Figures:

Series 10018-194:	<u>Tables 26 and 27, Figures 18 and 19</u>
Series 10018-198:	<u>Tables 28 and 29, Figures 20 and 21</u>
Series 11644-6:	<u>Tables 30 and 31, Figures 22 and 23</u>

Corresponding data for these three catalysts, or their simulants, are given in Tables 32 and 33 in bench-scale and MICTR equipment. In the bench-scale runs with decalin the Shell 113 prototype catalyst appeared somewhat more active and stable than UOP-R8 catalyst, but the Shell 146 catalyst was considerably

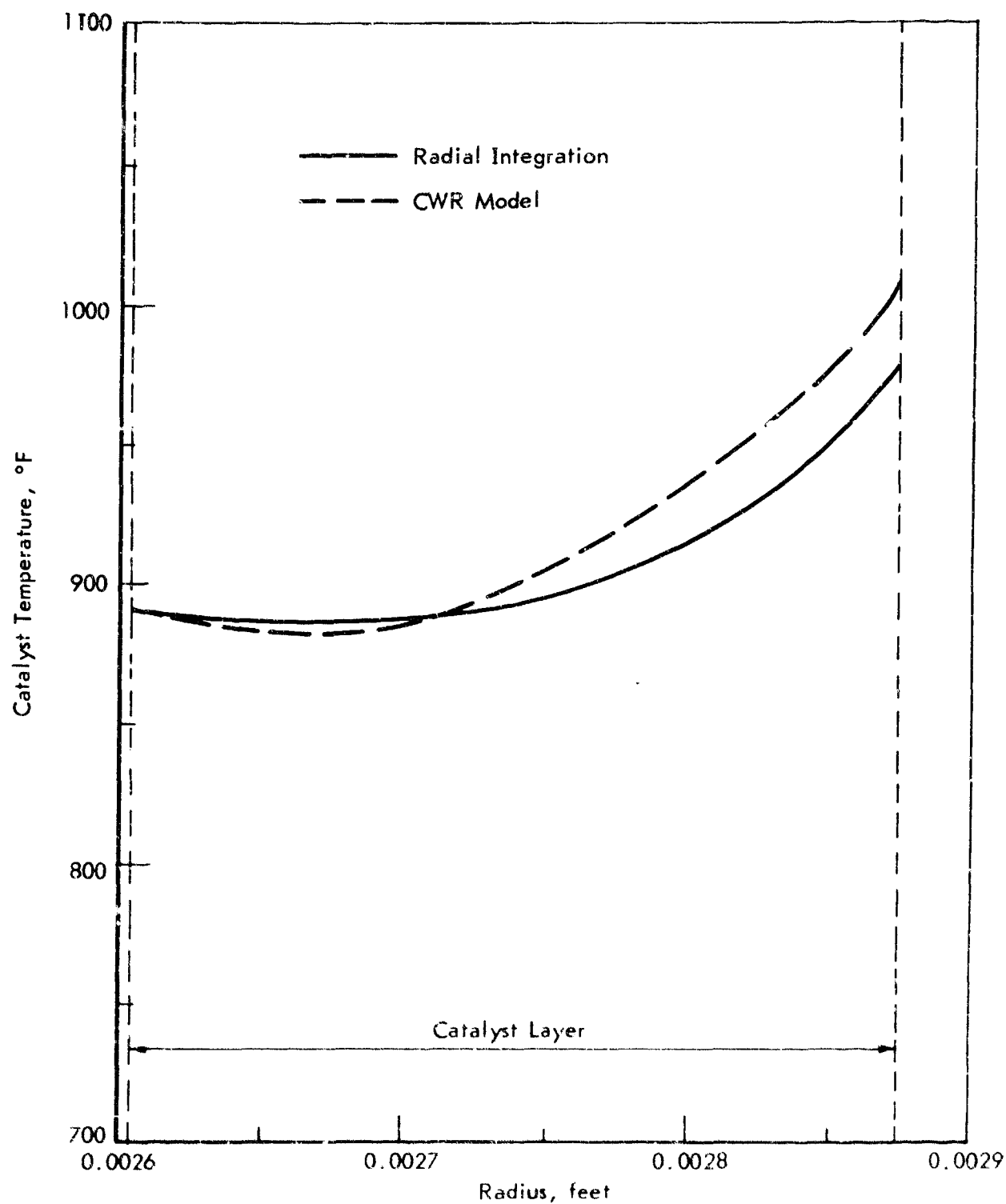


Figure 15. CALCULATED TEMPERATURE OF CATALYST LAYER

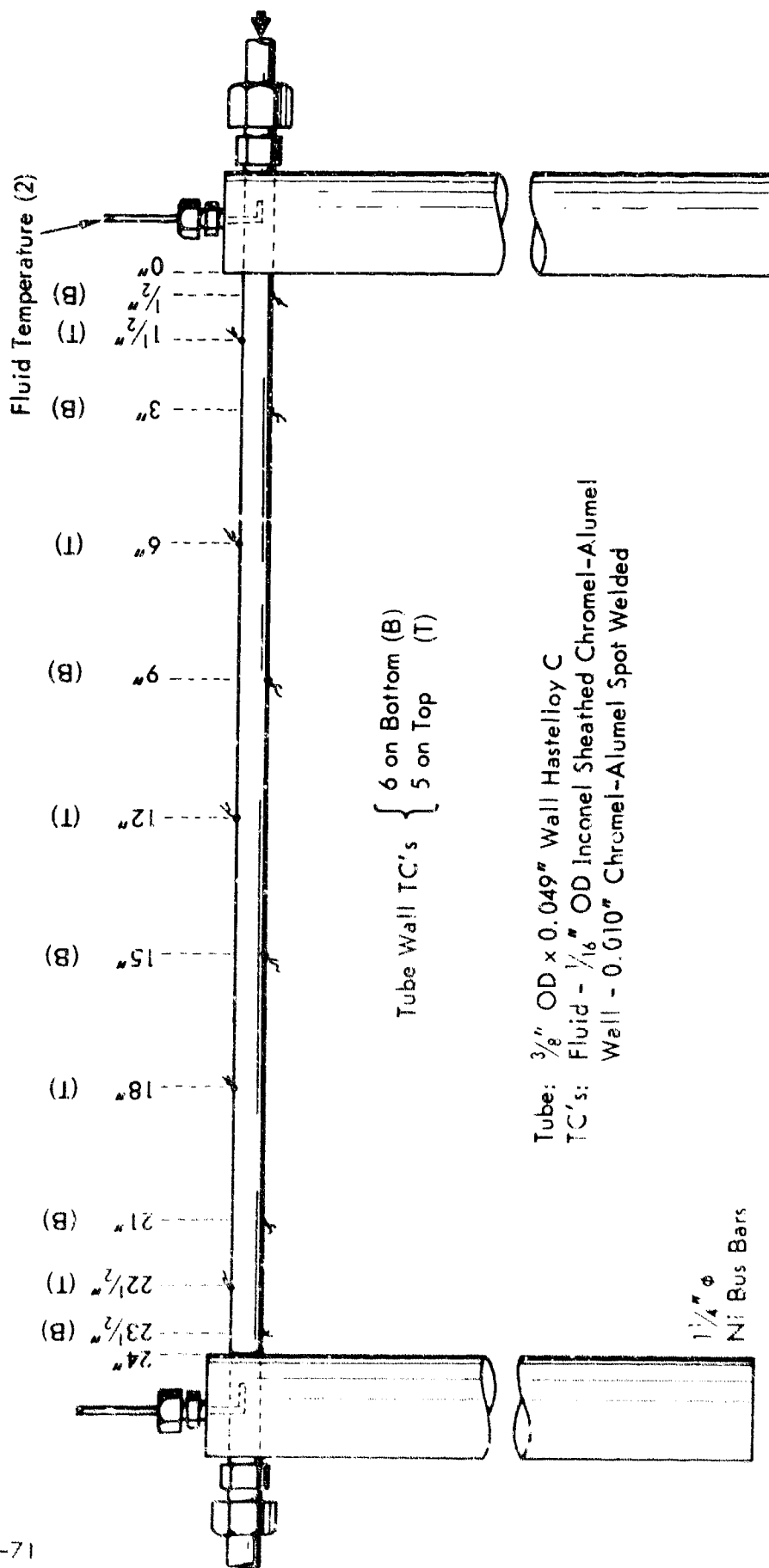


Figure 16. FSSTR REACTOR SECTION: 0.277" ID x 2-FT LENGTH

Table 25. FSSTR: DEHYDROGENATION OF DECALIN IN 2-FT REACTOR,  
SUMMARY OF TESTS WITH THREE CATALYST FORMULATIONS

Reactor: 3/8" O.D. x 0.049" wall x 2 ft long Hastelloy C

Catalysts: UOP-R8                      0.8% Pt on 1/16" Al<sub>2</sub>O<sub>3</sub> Spheres  
                   Shell 10280-113        4% Pt on 1/16" Al<sub>2</sub>O<sub>3</sub> Spheres  
                   Shell 10860-146        4% Pt on 10-20 mesh Al<sub>2</sub>O<sub>3</sub>

Nominal Inlet Conditions

Pressure        900 psig

Temperature    900°F

Feed Rate       25.0 lb/hr (545 LHSV)

Feed: 33.6% t-DHN

65.8% C-DHN

0.5% THN

0.1% other

Test Series (Catalyst)	Time, Hr:Min	Heat,		Outlet Fluid, °F	DHN Conv'n, %	Selectivity to	
		Btu (hr/ft <sup>2</sup> )	Btu lb			THN	N
10018-194 (UOP-R8)	11:15 - 11:50	-1,400	-8	723	13.9	85.2	14.8
	12:05 - 12:50	43,200	251	801	32.0	63.1	36.9
	13:10	106,800	620	906	54.5	31.5	68.0
	13:40	"	"	924	52.5	29.2	70.2
	13:50	139,100	807	1001	62.4	17.0	81.4
	14:15	"	"	1038	59.0	16.1	82.2
	14:40	"	"	1051	57.2	16.5	81.8
	15:00 - 15:35	22,600	131	795	20.1	67.9	32.1
10018-196 (Shell 10280-113)	10:35 - 11:05	-1,300	-8	720	14.0	83.8	16.2
	11:25 - 12:05	47,000	273	805	33.6	61.4	38.6
	12:20	104,500	606	927	51.3	28.7	71.3
	13:20	"	"	1000	43.7	21.7	78.3
	14:20	"	"	1054	39.1	19.6	80.4
	14:50 - 15:45	16,300	94	800	14.5	61.4	38.6
11644-6 (Shell 10860-146)	10:35 - 10:45	-1,300	-7	709	16.9	89.8	10.2
	11:10 - 12:15	75,600	439	800	53.0	59.9	40.1
	12:30	150,100	871	916	78.3	22.0	78.0
	13:30	"	"	942	73.3	19.9	80.1
	13:50	176,100	1022	1009	80.7	10.4	89.6
	14:50	"	"	1035	78.3	9.5	90.5
	15:50	"	"	1050	77.3	8.9	91.1
	16:10 - 16:35	38,300	222	794	29.9	66.5	33.5

Table 26 and Figure 17 follow

Table 26. FSSTR: DEHYDROGENATION OF DECALIN OVER UOP-R8 IN 2-FT REACTOR,  
DATA SUMMARY SERIES 10018-194

Reactor No. 10018-49; 0.277" ID x 0.049" Wall x 2 Ft Long Hastelloy C

Feed: 99.5% Decalin, 0.5% Tetralin;  
25.0 lb/hr, 545 LHSV, 59,740 lb/(hr · ft<sup>2</sup>)

Run No.	Experimental Data										Smoothed and Calculated Data						
	Fluid Temp., °F		Pressure, psig		Decalin Conv'n %	Selectivity, %		Tube Wall T.C.'s		Temp., °F	Tube length, in.	Wall Temp., °F <sup>a)</sup>		Heat Flux, d <sup>b)</sup> Btu hr · ft <sup>2</sup> × 10 <sup>-3</sup>	Cumulative Heat <sup>c)</sup>		
	In	Out	In	Out		to Tetralin	to Naphthalene	Location				Outside	Inside		Btu hr	Btu lb	
								Inches <sup>d)</sup>	Position <sup>e)</sup>								
1150	901	725	900	884	15.9	85.2	14.8	0.5	B	842	0	(860)	(861)	-1.7	0	0.0	
(Fresh catalyst charge. Activated 1 hr. in H <sub>2</sub> at 1100°F.)																	
								1.5	T	850	2	813	814	-1.6	-38	-1.5	
								3	B	798	6	777	774	-1.5	-57	-2.5	
								6	T	769	10	755	754	-1.4	-92	-3.7	
								9	B	760	14	742	743	-1.4	-126	-5.0	
								12	T	745	18	735	736	-1.4	-162	-6.4	
								15	B	744	22	730	731	-1.4	-193	-7.7	
								18	T	732	24	(728)	(729)	-1.4	-210	-8.4	
								21	B	735	(0-24)			(-1.45)			
								22.5	T	730							
								23.5	B	731							
1250	905	801	898	866	32.0	65.1	36.9	0.5	B	893	0	(905)	(896)	45.1	0	0	
								1.5	T	887	2	868	859	45.1	521	20.8	
								3	B	859	6	841	832	45.2	1565	62.6	
								6	T	844	10	841	832	45.2	2610	104.4	
								9	B	845	14	845	834	45.2	3654	146.2	
								12	T	845	18	846	837	45.2	4699	188.0	
								15	B	845	22	850	841	45.2	5745	229.7	
								18	T	845	24	(852)	(845)	45.2	6265	250.6	
								21	B	853	(0-24)			(45.2)			
								22.5	T	849							
								23.5	B	850							
1310	900	906	899	853	54.5	31.5	68.0	0.5	B	953	0	(956)	(934)	106.8	0	0	
								1.5	T	950	2	949	947	106.8	1291	51.7	
								3	B	946	6	946	924	106.9	2874	155.0	
								6	T	944	10	958	936	106.8	6457	258.3	
								9	B	956	14	970	948	106.8	9038	361.5	
								12	T	969	18	985	961	106.8	11620	464.0	
								15	B	980	22	997	975	106.7	14200	568.0	
								18	T	980	24	(1003)	(981)	106.7	15490	619.6	
								21	B	997	(0-24)			(106.8)			
								22.5	T	994							
								23.5	B	999							
1340	903	924	899	853	52.5	29.2	70.2	0.5	B	955	0	(956)	(934)	106.8	0	0	
(Continuation of Run 1310)																	
								1.5	T	950	2	949	927	106.8	1291	51.7	
								3	B	946	6	946	924	106.9	2874	155.0	
								6	T	943	10	959	937	106.8	6457	258.3	
								9	B	956	14	974	952	106.8	9038	361.5	
								12	T	968	18	990	968	106.7	11620	464.0	
								15	B	982	22	1006	984	106.7	14200	567.9	
								18	T	986	24	(1015)	(993)	106.7	15490	619.5	
								21	B	1006	(0-24)			(106.8)			
								22.5	T	1007							
								23.5	B	1014							
1350	897	1001	899	845	62.4	17.0	81.4	0.5	B	989	0	(987)	(958)	139.2	0	0	
								1.5	T	984	2	988	959	139.2	1683	67.5	
								3	B	992	6	997	968	139.2	5048	201.9	
								6	T	993	10	1016	988	139.2	8413	336.5	
								9	B	1012	14	1039	1011	139.1	11780	471.0	
								12	T	1031	18	1065	1037	139.0	15140	605.5	
								15	B	1050	22	1095	1068	138.9	18500	739.9	
								18	T	1061	24	(1111)	(1084)	138.9	20180	807.1	
								21	B	1091	(0-24)			(139.1)			
								22.5	T	1095							
								23.5	B	1106							
1415	900	1058	900	846	59.0	16.1	82.2	0.5	B	995	0	(989)	(960)	139.2	0	0	
(Continuation of Run 1350)																	
								1.5	T	987	2	990	961	139.2	1683	67.5	
								3	B	996	6	999	970	139.2	5048	201.9	
								6	T	996	10	1020	992	139.1	8412	336.5	
								9	B	1016	14	1050	1022	139.1	11780	471.0	
								12	T	1038	18	1082	1055	139.0	15140	605.5	
								15	B	1061	22	1121	1094	138.9	18400	739.8	
								18	T	1078	24	(1142)	(1116)	138.8	20170	806.9	
								21	B	1112	(0-24)			(139.1)			
								22.5	T	1124							
								23.5	B	1140							
1440	902	1051	900	848	57.2	16.5	81.8	0.5	B	992	0	(983)	(959)	139.2	0	0	
(Continuation of Run 1350)																	
								1.5	T	984	2	985	960	139.2	1683	67.5	
								3	B	995	6	998	969	139.2	5048	201.9	
								6	T	994	10	1019	991	139.2	8413	336.5	
								9	B	1014	14	1050	1022	139.1	11780	471.0	
								12	T	1038	18	1082	1055	139.0	15140	605.5	
								15	B	1063	22	1130	1103	138.9	18400	739.7	
								18	T	1082	24	(1152)	(1126)	138.8	20170	806.9	
								21	B	1120	(0-24)			(139.1)			
								22.5	T	1134							
								23.5	B	1150							
1530	910	795	890	844	20.1	67.9	34.1	0.5	B	876	0	(894)	(887)	22.4	0	0	
								1.5	T	866	2	845	840	22.4	271	10.9	
								3	B	852	6	805	800	22.6	818	32.7	
								6	T	837	10	798	795	22.7	1366	54.6	
								9	B	801	14	800	795	22.7	1914	76.6	
								12	T	797	18	807	800	22.6	2461	98.5	
								15	B	811	22	815	815	22.6	3000	120.5	
								18	T	805	24	(804)	(819)	22.6	3481	135.5	
								21	B	817	(0-24)			(22.6)			
								22.5	T	797							
								23.5	B	802							

- a) Tube temperatures to outside wall at indicated inches from inlet and bus bar.  
b) Location of T.C. junction on horizontal tube. B = Bottom, T = Top.  
c) Outside wall temperatures by smoothing experimental data. Inside temperatures by calculation.  
d) Corrected for length. Values in ( ) are average over entire heated length.  
e) Wet heat to fluid up to indicated tube length.

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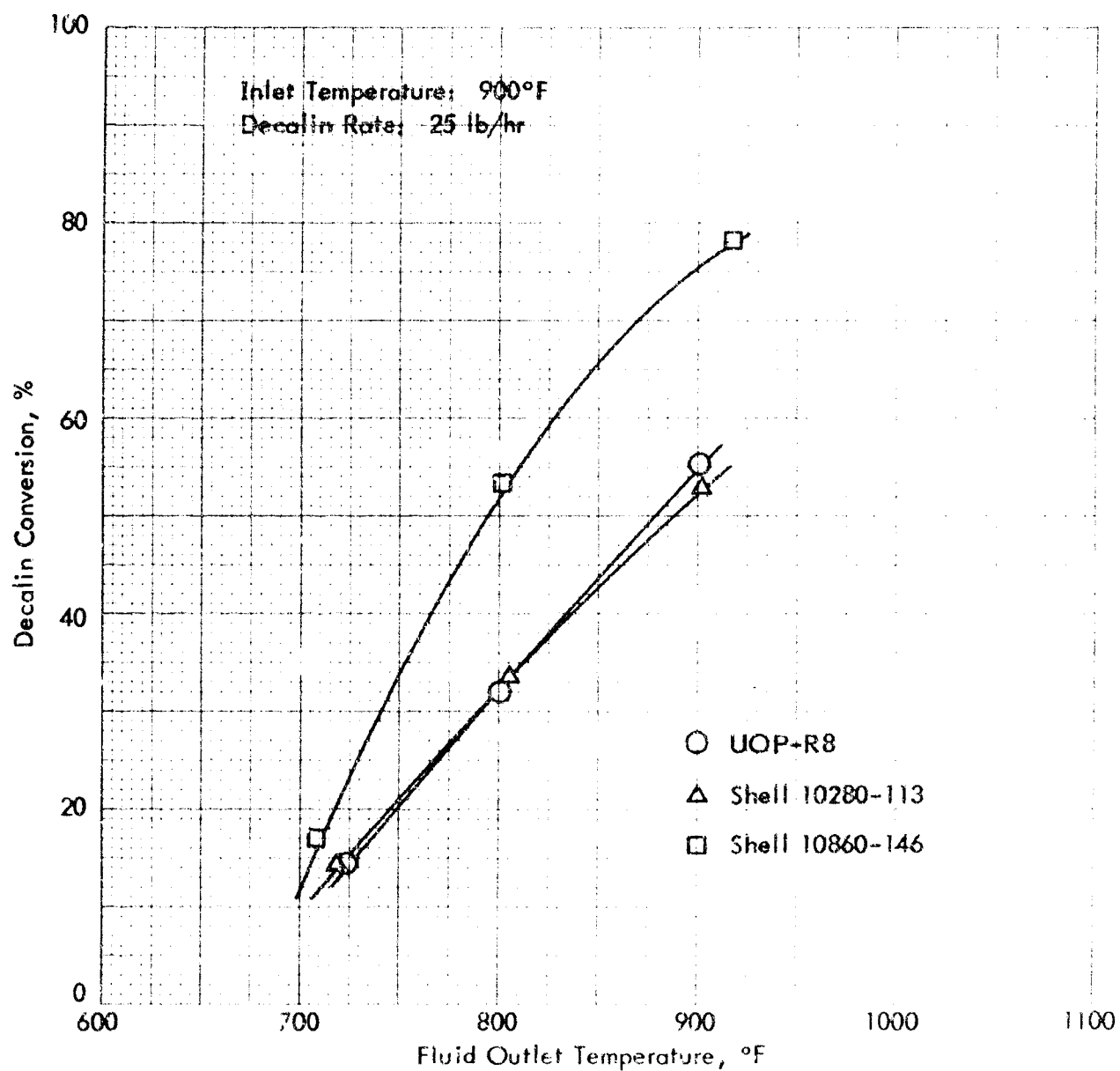


Figure 17. FSSTR: DEHYDROGENATION OF DECALIN IN 2-FT REACTOR,  
INITIAL ACTIVITY OF THREE CATALYST FORMULATIONS

Table 27. FSSTR: DEHYDROGENATION OF DECALIN OVER SHELL  
10860-146 IN 2-FT REACTOR, PRODUCT ANALYSES  
FOR SERIES 11644-6

Time	Product Composition, %w			
	t-Decalin	c-Decalin	Tetralin	Naphthalene
10:35 - 10:45 <sup>1)</sup>	51.6	31.6	15.0	1.6
11:10 - 12:15 <sup>1)</sup>	35.7	12.5	31.6	20.2
12:30 <sup>2)</sup>	17.7	5.1	17.8	59.4
13:30 <sup>2)</sup>	20.8	7.1	15.1	57.0
13:50 <sup>2)</sup>	15.1	5.3	9.0	70.6
14:50 <sup>2)</sup>	16.3	6.5	8.0	69.2
15:50 <sup>2)</sup>	16.8	7.1	7.4	68.7
16:10 - 16:35 <sup>1)</sup>	41.6	29.5	19.5	9.5
Feed	33.6	65.9	0.5	

1) Average values over indicated time period.

2) Smoothed data.

Figures 18 and 19 and Table 28 follow

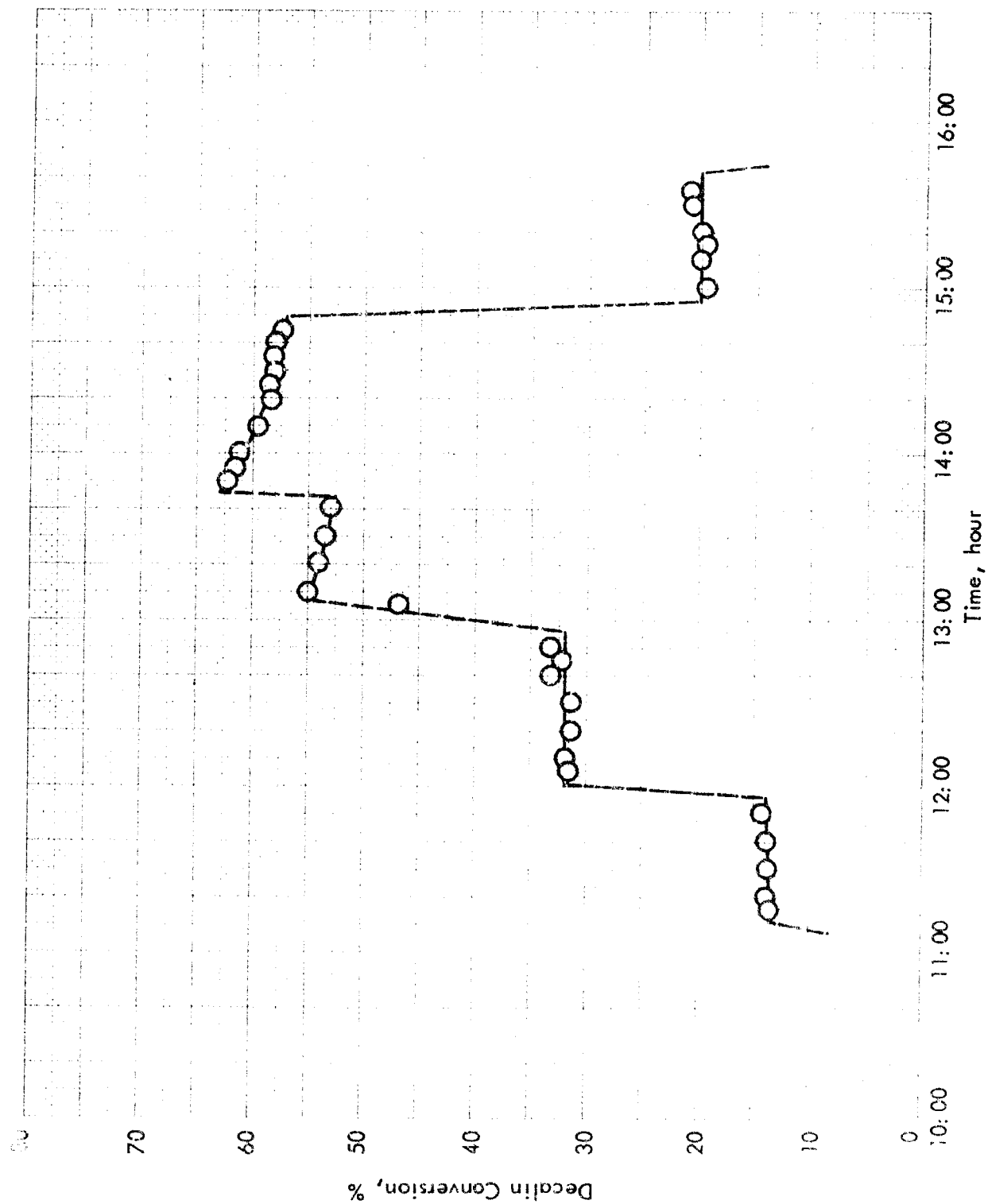


Figure 18. FSSTR: DEHYDROGENATION OF DECALIN OVER UOP-R8 IN 2-FT REACTOR,  
DECALIN CONVERSION, SERIES 10018-194

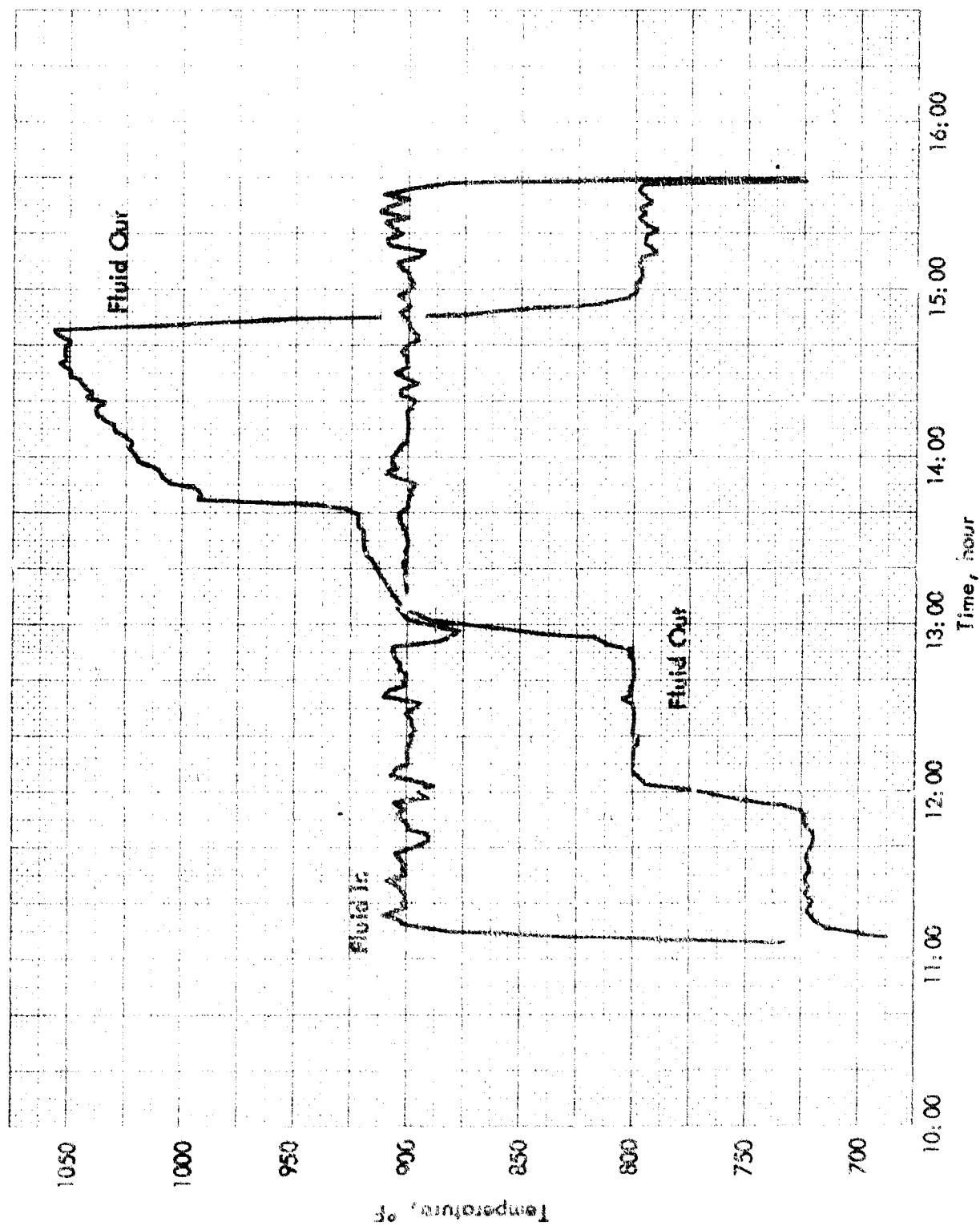


Figure 19. FSSTR: DEHYDROGENATION OF DECALIN OVER UOP-R8 IN 2-FT REACTOR,  
 FLUID TEMPERATURES, SERIES 10018-194

**Table 28. FSSTR: DEHYDROGENATION OF DECALIN OVER SHELL 10280-113  
IN 2-FT REACTOR, DATA SUMMARY SERIES 10018-198**

Reactor No. 10018-49; 0.277" ID x 0.049" Wall x 2 Ft Long Hastelloy C

Feed: 99% Decalin, 0.5% Tetralin;  
25.0 lb/hr, 545 LHSV, 59,740 lb/(hr · ft<sup>2</sup>)

Run No. 10018- 198-	Experimental Data										Smoothed and Calculated Data					
	Fluid Temp., °F		Pressure, psig		Decalin Conv'n %	Selectivity, %		Tube Wall T.C.'s			Tube Length, In.	Well Temp., °F <sup>c)</sup>		Heat Flux, <sup>d)</sup> Btu (hr · ft <sup>2</sup> ) × 10 <sup>-3</sup>	Cumulative Heat <sup>e)</sup>	
	In	Out	In	Out		to Tetra lin	to Naphthalene	Location		Temp. °F		Outside	Inside		Btu hr	Btu lb
								Inches <sup>a)</sup>	Position <sup>b)</sup>							
1100	906	726	900	895	14.0	85.8	16.2	0.5 1.5 3 6 9 12 15 18 21 22.5 23.5	B T B T B T B T B T B	850 849 800 771 760 743 742 731 731 728 730	0 2 6 10 14 18 22 24 (0-24)	(895) (821) (775) (754) (741) (733) (729) (728) (729)	-1.6 -1.5 -1.5 -1.5 -1.5 -1.5 -1.2 -1.2 (-1.31)	0 -18 -52 -84 -115 -145 -175 -190	0 -0.7 -9.1 -3.4 -4.6 -5.8 -7.0 -7.6	
1200	895	805	898	865	33.6	61.4	38.6	0.5 1.5 3 6 9 12 15 18 21 22.5 23.5	B T B T B T B T B T B	900 898 857 856 849 848 850 847 854 855 856	0 2 6 10 14 18 22 24 (0-24)	(924) (883) (856) (849) (848) (850) (854) (856) (846)	46.9 47.0 47.1 47.1 47.1 47.1 47.1 47.1 (47.0)	0 568 1704 2842 3980 5117 6255 6844	0 22.7 68.2 113.7 159.2 204.7 250.2 272.9	
1220	905	927	896	852	51.3	28.7	71.3	0.5 1.5 3 6 9 12 15 18 21 22.5 23.5	B T B T B T B T B T B	974 967 956 956 956 962 963 980 990 1007 1020 1023	0 2 6 10 14 18 22 24 (0-24)	(989) (962) (956) (956) (956) (974) (972) (1014) (1026) (1005)	104.4 104.5 104.5 104.5 104.5 104.4 104.4 104.4 (104.5)	0 1265 3790 6516 8842 11370 13690 15150	0 50.5 151.6 252.6 353.7 454.7 555.7 606.1	
1320	894	1000	896	852	43.7	21.7	78.7	0.5 1.5 3 6 9 12 15 18 21 22.5 23.5	B T B T B T B T B T B	968 965 958 958 957 966 966 1008 1029 1052 1078 1087	0 2 6 10 14 18 22 24 (0-24)	(973) (961) (962) (954) (1000) (1032) (1051) (1092) (1072)	104.5 104.5 104.5 104.5 104.4 104.5 104.2 104.2 (104.4)	0 1255 3790 6516 8840 11360 13890 15140	0 50.5 151.6 252.6 353.6 454.5 555.4 605.8	
1420	897	1054	896	852	39.1	19.6	80.4	0.5 1.5 3 6 9 12 15 18 21 22.5 23.5	B T B T B T B T B T B	974 969 965 970 987 1004 1036 1062 1095 1123 1134	0 2 6 10 14 18 22 24 (0-24)	(981) (966) (970) (970) (1021) (1065) (1115) (1140) (1110)	104.5 104.5 104.5 104.4 104.4 104.5 104.1 104.1 (104.4)	0 1265 3789 6514 8838 11360 13880 15140	0 50.5 151.6 252.6 353.5 454.4 555.2 605.5	
1540	896	800	895	868	14.5	61.4	38.6	0.5 1.5 3 6 9 12 15 18 21 22.5 23.5	B T B T B T B T B T B	872 868 855 810 800 795 800 801 814 817 821	0 2 6 10 14 18 22 24 (0-24)	(920) (851) (807) (798) (798) (804) (816) (821) (821)	16.0 16.2 16.3 16.3 16.3 16.3 16.3 16.2 (16.3)	0 195 507 981 1375 1769 2162 2559	0 7.8 23.5 59.2 95.0 120.8 166.5 194.5	

- a) T.C.'s spotwelded to outside wall at indicated inches from inlet and bus bar.  
b) Location of T.C. junction on horizontal tube. B = Bottom, T = Top.  
c) Outside wall temperatures by smoothing experimental data. Inside temperatures by calculation.  
d) Corrected for losses. Values in ( ) are average over entire heated length.  
e) Not heat to fluid up to indicated tube length.

Table 29. FSSTR: DEHYDROGENATION OF DECALIN OVER  
SHELL 10280-113 IN 2-FT REACTOR,  
PRODUCT ANALYSES FOR SERIES 10018-198

Time	Product Composition, %w			
	t-Decalin	c-Decalin	Tetralin	Naphthalene
10:35 - 11:05 <sup>1)</sup>	43.0	43.2	11.7	2.1
11:25 - 12:05 <sup>1)</sup>	35.7	31.6	20.5	12.2
12:20 <sup>2)</sup>	26.1	24.0	15.0	34.9
13:20 <sup>2)</sup>	26.8	30.9	9.8	32.5
14:20 <sup>2)</sup>	27.2	35.0	8.0	29.8
14:50 - 15:45 <sup>1)</sup>	35.8	50.0	9.0	5.2
Feed	33.6	65.9	0.5	

1) Average values over indicated time period.

2) Smoothed data.

Figures 20 and 21 and Table 30 follow

**Table 30. FSSTR: DEHYDROGENATION OF DECALIN OVER SHELL 10860-146  
IN 2-FT REACTOR, DATA SUMMARY SERIES 11644-6**

Reactor No. 10018-49; 0.277" ID x 0.049" Wall x 2 Ft Long Hastelloy C

Feed: 99.5% Decalin, 0.5% Tetralin;  
25.0 lb/hr, 545 LHSV, 59,740 lb/(hr · ft<sup>2</sup>)

Run No. 11644-6-	Experimental Data										Smoothed and Calculated Data						
	Fluid Temp., °F		Pressure, psig		Decalin Conv'n %	Selectivity, %		Tube Wall		Temp. °F	Tube Length, In.	Wall Temp., °F		Heat Flux, d <sup>1</sup> Btu (hr · ft <sup>2</sup> ) × 10 <sup>-3</sup>	Cumulative Heat <sup>2</sup>		
	In	Out	In	Out		to Tetralin	to Naphthalene	Location				Outside	Inside		Btu hr	Btu lb	
								Inches <sup>a</sup>	Posit								
1045	906	709	900	825	16.9	89.8	10.2	0.5 1.5 3 6 9 12 15 18 21 22.5 25.5	B T B T B T B T B T B	0 2 4 7 10 14 18 22 24 24 (-24)	(900) 782 785 730 722 718 716 717 (716) (717)	(901) 783 746 731 725 719 717 (717)	-1.6 -1.4 -1.3 -1.2 -1.2 -1.2 -1.2 -1.2 -1.2 (-1.26)	0 -17 -49 -80 -109 -139 -168 -185	0 -0.7 -2.0 -3.2 -4.4 -5.6 -6.7 -7.5		
1200	900	800	900	795	51.0	90.9	8.0	3 6 9 12 15 18 21 22.5 25.5	B T B T B T B T B	0 2 6 10 14 18 22 24 (0-24)	(980) 882 845 842 847 857 871 871 (878)	(965) 866 828 825 830 840 855 (862)	75.3 75.5 75.6 75.6 75.6 75.6 75.6 75.6 (75.6)	0 912 2740 4568 6596 8224 10050 10970	0 36.5 109.6 182.7 255.9 329.0 402.1 458.6		
1250	904	916	902	741	78.5	22.0	78.0	0.5 1.5 3 6 9 12 15 18 21 22.5 25.5	B T B T B T B T B T B	0 2 6 10 14 18 22 24 (0-24)	(1050) 980 942 942 943 959 985 1019 (1053)	(1020) 942 910 911 927 954 988 (1008)	149.9 150.1 150.2 150.2 150.1 150.0 150.0 (150.1)	0 1815 5445 9075 12700 16550 19960 21770	0 72.5 217.7 362.9 508.1 655.2 798.3 870.8		
1350	899	942	902	741	75.3	19.9	80.1	0.5 1.5 3 6 9 12 15 18 21 22.5 25.5	B T B T B T B T B T B	0 2 6 10 14 18 22 24 (0-24)	(1050) 979 942 942 941 955 985 1000 (1064)	(1020) 942 910 914 936 969 1011 (1054)	149.9 150.1 150.2 150.2 150.1 150.0 149.9 (150.1)	0 1815 5445 9075 12700 16550 19950 21770	0 72.5 217.7 362.9 508.1 655.2 798.2 870.7		
1350	899	1009	905	718	30.7	10.4	89.6	0.5 1.5 3 6 9 12 15 18 21 22.5 25.5	B T B T B T B T B T B	0 2 6 10 14 18 22 24 (0-24)	(1070) 997 975 978 1005 1049 1107 (1139)	(1035) 961 936 941 969 1014 1075 (1105)	176.0 176.2 176.2 176.2 176.1 175.9 175.8 (176.1)	0 2129 6588 10650 14910 19170 23480 25550	0 85.1 255.5 426.0 596.5 766.6 936.8 1021.8		
1450	900	1055	905	718	78.5	9.5	90.5	0.5 1.5 3 6 9 12 15 18 21 22.5 25.5	B T B T B T B T B T B	0 2 6 10 14 18 22 24 (0-24)	(1070) 1005 975 978 1005 1062 1124 (1160)	(1035) 965 937 947 969 1027 1090 (1127)	176.0 176.2 176.2 176.2 176.1 175.9 175.8 (176.1)	0 2129 6588 10650 14910 19160 23480 25550	0 85.1 255.5 425.9 596.5 766.6 936.7 1021.7		
1550	901	1050	905	718	77.5	8.9	91.1	0.5 1.5 3 6 9 12 15 18 21 22.5 25.5	B T B T B T B T B T B	0 2 6 10 14 18 22 24 (0-24)	(1070) 1005 975 978 1005 1062 1124 (1179)	(1035) 967 948 952 969 1027 1090 (1146)	176.0 176.2 176.2 176.2 176.1 175.8 175.7 (176.1)	0 2129 6588 10650 14910 19160 23480 25550	0 85.1 255.5 425.9 596.5 766.6 936.6 1021.6		
1550	900	704	901	825	29.9	66.5	55.5	0.5 1.5 3 6 9 12 15 18 21 22.5 25.5	B T B T B T B T B T B	0 2 6 10 14 18 22 24 (0-24)	(950) 845 795 797 795 780 805 809 (845)	(944) 845 795 778 776 802 821 (845)	58.0 58.2 58.4 58.4 58.5 58.5 58.5 (58.5)	0 461 587 654 687 707 707 (707)	0 184.4 257.6 327.6 397.6 467.7 537.7 607.7		

a) T.C.'s attached to outside wall at indicated inches from inlet and header.  
b) Location of T.C. junction on horizontal tube. B = Bottom, T = Top.  
c) Outside wall temp. returns by smoothing experimental data. Inside temperatures by calculation.  
d) Corrected for losses. Values in ( ) are average over entire heated length.  
e) Net heat to fluid up to indicated tube length.

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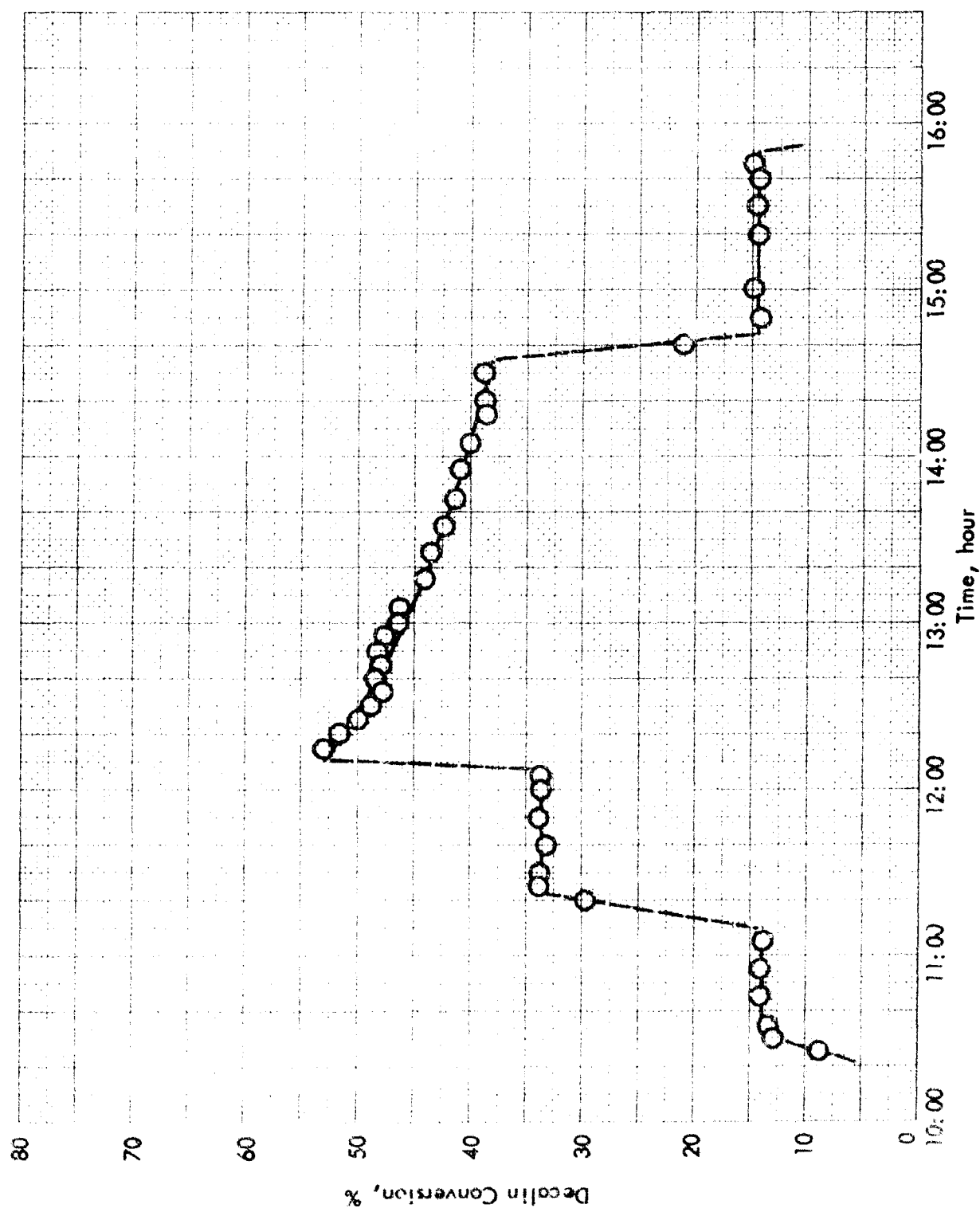


Figure 20. FSSTR: DEHYDROGENATION OF DECALIN OVER SHELL 10280-113 IN 2-FT REACTOR,  
DECALIN CONVERSION, SERIES 10018-198

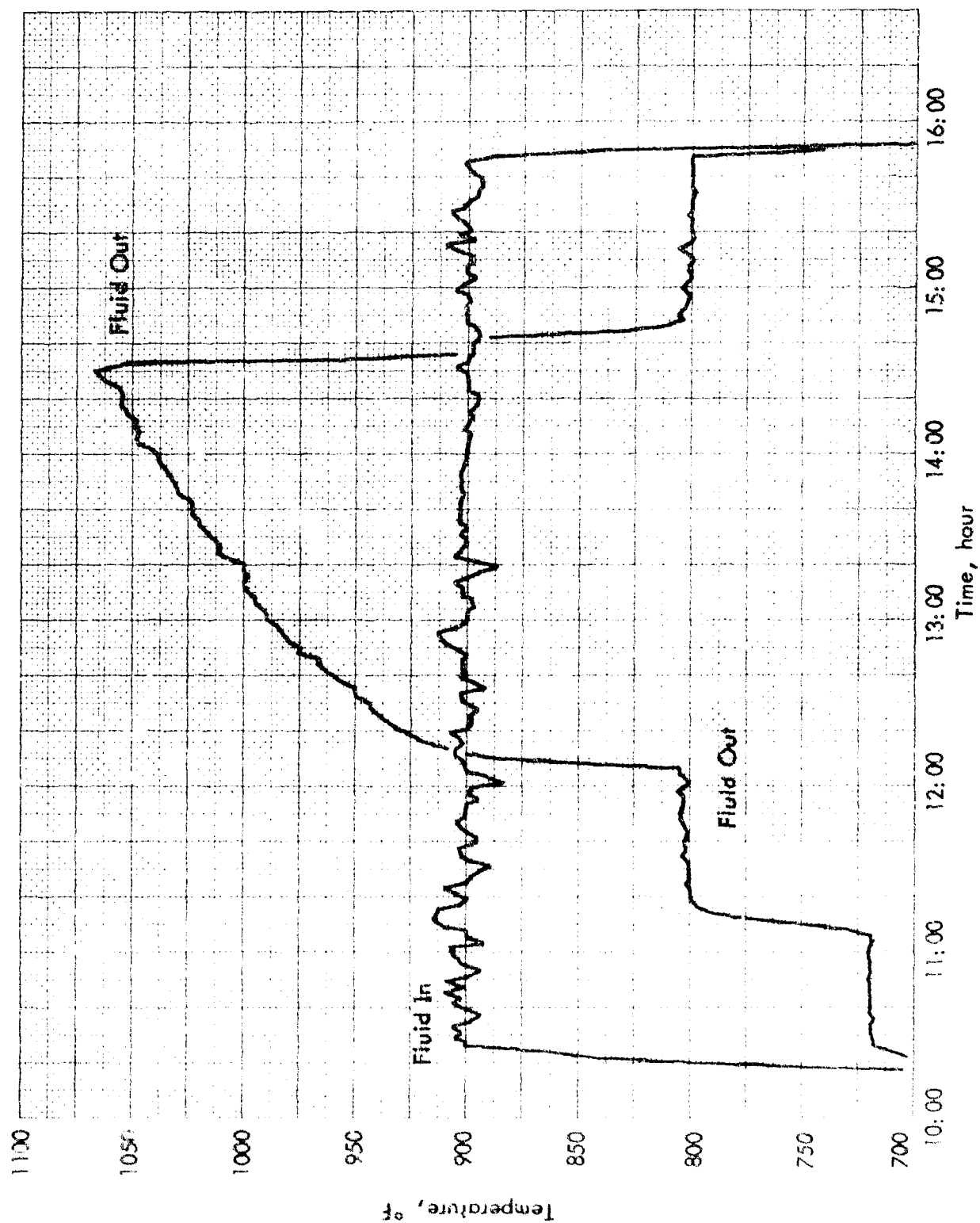


Figure 21. FSSTR: DEHYDROGENATION OF DECALIN OVER SHELL 10280-113 IN 2-FT REACTOR, FLUID TEMPERATURES, SERIES 10018-198

Table 31. FSSTR: DIHYDROGENATION OF DECALIN OVER UOP-R8  
IN 2-FT REACTOR, PRODUCT ANALYSES FOR SERIES 10018-194

Time	Product Composition, %w				
	t-Decalin	c-Decalin	Tetralin	Naphthalene	Unknown
11:15 - 11:50 <sup>1)</sup>	43.4	42.9	11.8	1.9	0
12:05 - 12:50 <sup>1)</sup>	36.7	32.1	20.1	11.1	0
13:10 <sup>2)</sup>	26.0	20.9	17.4	35.4	0.3
13:40 <sup>2)</sup>	26.6	22.3	15.6	35.2	0.3
13:50 <sup>2)</sup>	21.1	18.0	11.0	48.9	1.0
14:15 <sup>2)</sup>	22.0	20.5	9.9	46.6	1.0
14:40 <sup>2)</sup>	22.6	21.7	9.8	44.8	1.0
15:00 - 15:35 <sup>1)</sup>	38.8	41.5	13.6	6.0	0
Feed	33.6	65.9	0.5		

1) Average values over indicated time period.

2) Smoothed data.

Figures 22 and 23 follow

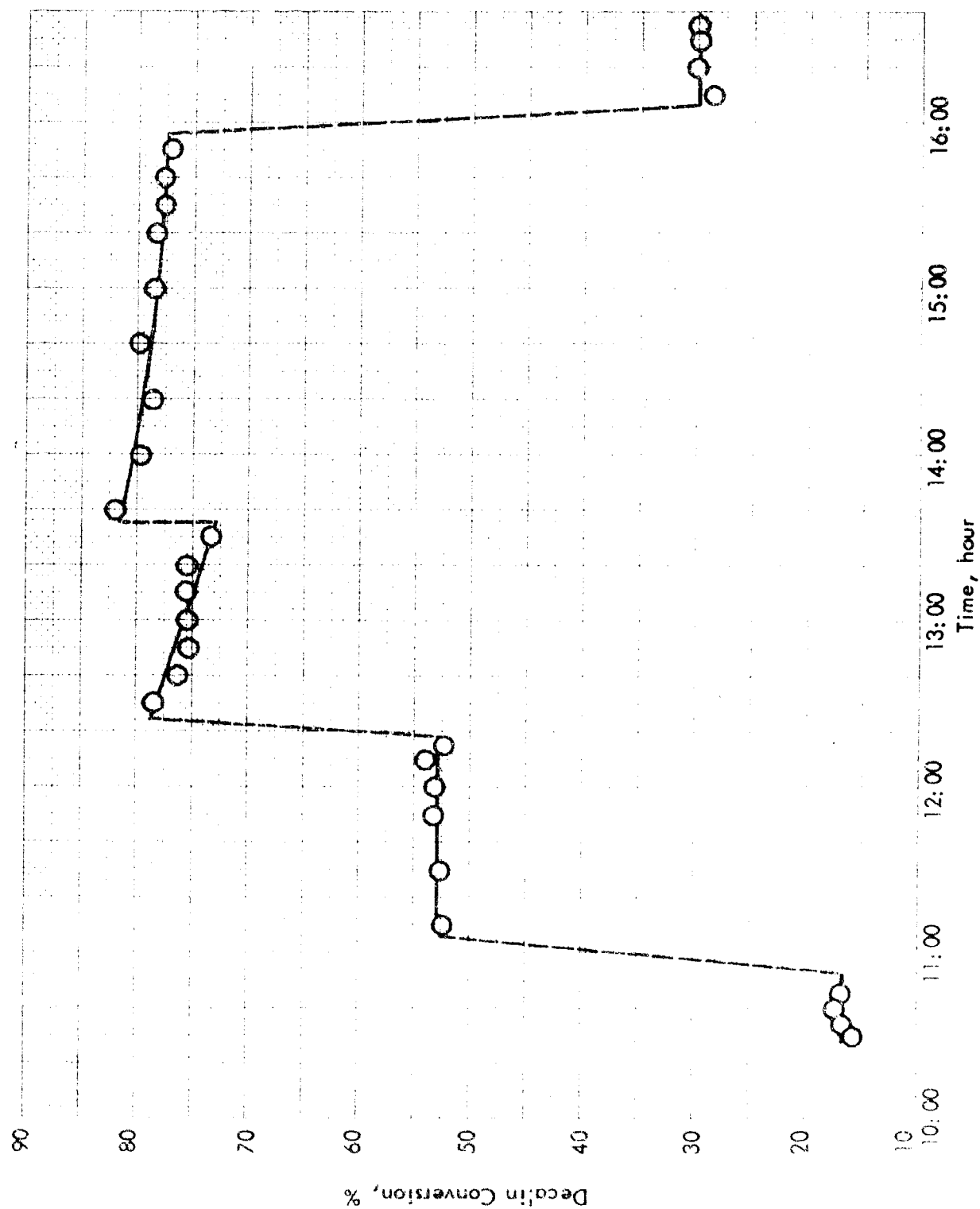


Figure 22. FSSTR: DEHYDROGENATION OF DECALIN OVER SHELL 10860-146 IN 2-FT REACTOR,  
DECALIN CONVERSION, SERIES 11644-6

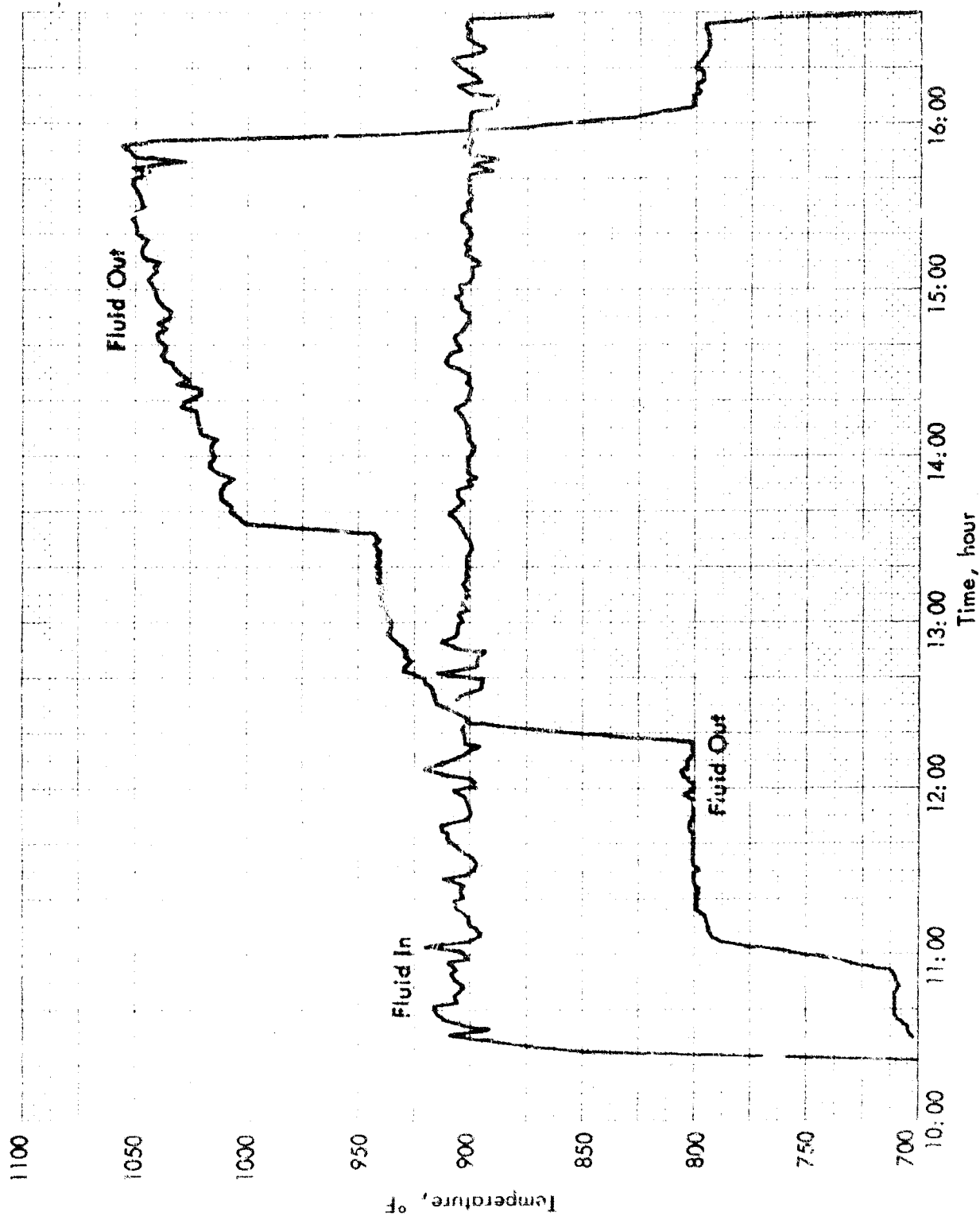


Figure 23. FSSTR: DEHYDROGENATION OF DECALIN OVER SHELL 10860-146 IN 2-FT REACTOR,  
FLUID TEMPERATURES, SERIES 11644-6

Table 32. DEHYDROGENATION OF DECALIN OVER VARIOUS CATALYSTS

Feed: F-113 DHN

Pressure: 10 atm

Feed Composition:

LHSV: 100

25.0% trans DHN

Reaction Period: 30 minutes

74.5% cis DHN

0.4% THN

Reactor: Bench Scale, 5" bed in 1/2" IPS SS tubing furnace heated.

Catalyst	% DHN Conversion at, °F					ΔT, °F of Catalyst Bed at, °F				
	842	932	1022	1112	1202	842	932	1022	1112	1202
10860-146	43.7	58.3	62.2	71.2	84.1 <sup>a)</sup>	3	5	13	18	66 <sup>a)</sup>
UOP-R8	36.2	43.9	47.7 <sup>b)</sup>	-	-	14	43	160 <sup>b)</sup>	-	-
10280-107B <sup>c)</sup>	39.1	46.7	54.2	61.5	66.5	11	25	34	56	160
10280-91A <sup>c)</sup>	39.0	46.1	54.9	61.6	68.8	13	22	36	50	205

a) Reaction time: 10 minutes.

b) Catalyst completely deactivated at the end of this run.

c) 10280-113 prototype.

Table 33. ACTIVITIES OF CATALYSTS 10280-113 AND 10860-146 FOR METHYLCYCLOHEXANE DEHYDROGENATION AT SEVERAL TEMPERATURES

Conditions: MCH, LHSV 100, 10 atm pressure, furnace heated

Reactor: MICTR 4-1/4" bed in 1/4" O.D. S.S. Tubing, catalyst diluted 0.9/1.1 with quartz chips

Catalyst No.	% Pt	Support		Average Conversion of MCH to Toluene, %			
		Type	Mesh Size	Run No.	662	752	842°F
10860-70	1	UOP-R8 type Al <sub>2</sub> O <sub>3</sub>	~10	1156	24	45	71
10280-113	4	1	9-16	1108	20	43	69
10860-146	4	1	10-20	1177	53	59	86

better than either the UOP-R8 or Shell 113 catalysts. Thus the FSSTR data confirms these findings in general. The lesser activity/stability of the Shell 113 catalyst compared to UOP-R8 catalyst in the FSSTR compared to the results with the prototypes may be due to some misadventure in the makeup of the large batch prepared for FSSTR evaluation; this will be rechecked. In the MICTR the comparison involves MCH rather than decalin, but the order of activity bears out the results found in the FSSTR with decalin.

#### THERMAL STABILITY OF FUELS

Thermal stability studies have progressed along several lines during the past year. One of the chief advances has been in the completion of the prototype beta-ray backscatter rig for measuring deposits. This device, although still undergoing evaluation and testing, has been used successfully on standard ASTM, SD/M-7, and JFTOT Coker tubes. It has also been used in measuring the deposit thicknesses on titanium strips from experimental fuel tanks at Boeing Aircraft Company.

The characterization of candidate fuels requires the development of suitable apparatus to characterize routinely the behavior of the fuel at high temperature and pressure in both the absence and presence of endothermic reaction. To provide a method of so characterizing fuels, we developed and put into operation during the last contract period a piece of equipment, the Catalyst and Fuel System Test Rig (CAFSTR), capable of evaluating a fuel/catalyst combination to a maximum temperature of about 1300°F. However, a major difficulty in the utilization of this apparatus was the determination of the amount of deposit on the heating tubes, which, because of the high temperatures involved, could not be fabricated of aluminum and rated visually as is done with tubes from the ASTM Coker. Now that the beta-ray backscatter device is available, we shall be able to start fuel evaluations with the CAFSTR, in which Inconel 600 heater tubes are used. Because of color changes of the metal itself, color rating of the Inconel tubes is impossible and deposits from this equipment could not be evaluated in the past.

Further progress in thermal stability testing has been achieved, in that the JFTOT fuel tester is now complete and operative, and a number of tests have been made. The rig has been found to be delightfully simple and rapid to use, but does seem to have a high temperature limitation. This can probably be solved, however.

No further changes have been made on the SD/M-7 fuel coker, except that a Beckman Oxygen Analyzer has now been installed and is functioning properly.

Various thermal stability investigations have been made including brief studies of hydrocrackate fuels and current commercial Jet-A type fuels. In addition, we have been exploring a new class of potential additives for improving thermal stability of fuels. Several additives have been found to be effective, although tests to date have shown that they are beneficial only in fuels containing dissolved oxygen, the benefits from the additive being substantially the same as those from nitrogen sparging of the fuel. This suggests, of course, that the additives directly interfere with the degradation mechanisms in which oxygen is a participant.

### Measurement of Deposits by Beta-Ray Backscatter

After a broad investigation of possible ways to measure fuel coker deposits quantitatively, a prototype beta-ray backscatter test rig was built for this purpose. This equipment<sup>a)</sup> has been completed to an operational stage, and its characteristics and capabilities are now being evaluated. A photograph of the apparatus in its present state of development is shown in Figure 24. A description of the equipment appears in the Appendix.

Initially, the test rig was set up partially from ordered commercial and shop produced components, plus some borrowed components, including a vacuum pump and a scanning mechanism drive motor. These borrowed parts have now been replaced with a large capacity vacuum pump to provide rapid pumpdown, and a suitable drive motor, speed control and gear reduction arrangement to permit scanning of different sized tubes. In addition, the original temporary detector, devised by modifying one which had been used for other purposes, has been replaced by one designed specifically for this instrument, and embodying improvements based on experience with the first. A commercial detector which was ordered while the temporary one was used has finally arrived. Although this detector is no longer needed, we plan to test its operation in comparison with the newly designed one. However, we are not optimistic about the commercial unit, since it has a window thickness of approximately 2500 Å. Since it was ordered, we have determined that the maximum window thickness to avoid loss of sensitivity is about 1500 Å.

Our currently installed window has a thickness of approximately 1000 Å and has been in use 24 hours per day for five weeks without leakage. The differential pressure across the window, which is supported by a 280 line-per-inch ickel screen, is essentially one atmosphere, and the vacuum system holds readily at a few millimicrons Hg pressure. This clearly demonstrates that there are no holes in the window. However, the window has been a problem. If the window thickness is too small, the window either breaks or develops holes; if it is too thick, it cuts out the low energy electrons required for measurement of deposit thickness. We originally used a cellulose nitrate material which was on hand in the laboratory, but windows cast from this material, when sufficiently thin, proved too fragile and short-lived. The window material that we had planned to use (Union Carbide PARYLENE<sup>®</sup> film) was too thick (2600 Å). We have now received a thinner film (1000 Å PARYLENE<sup>®</sup> film), which we plan to install later. While we were waiting for delivery of the thin film, we successfully cast a 1000 Å film of PARLODIC<sup>®</sup> (also a cellulose nitrate material), which we are now using. Although the PARYLENE<sup>®</sup> material is believed to be stronger, the present window is performing satisfactorily and will not be replaced until necessary. The present arrangement of window film and wire screen permits about a 70 percent transmission of electrons, which is considered adequate.

A considerable amount of effort has been expended in the evaluation of this equipment and is still continuing. The effects of vacuum level, detector time constant, rotational-translational tube speed, detector head positioning, chart speed, pen response time, and a number of other variables have been under investigation.

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a) A. Telfer and R. M. Curtis of our Analytical Department were responsible for the design, construction and preliminary testing of this equipment.

Figure 24 follows



As was noted previously<sup>3</sup>, it is possible to reach saturation for any given electron energy level, and hence to reach a maximum deposit thickness beyond which readings will be inaccurate. If thicker deposits are to be measured, a dual set of detectors and sources might be required to be assured that true deposit thicknesses are being measured. In addition, the span of the current 5 mv single channel 5-inch strip recorder is so narrow that it cannot read out the signal from all deposit levels at the same sensitivity setting. This often requires multiple runs at different sensitivities in order to measure the entire deposit profile. This could be corrected by utilizing a 1 mv 10-inch strip recorder with dual channels. This would increase the sensitivity, and the use of the two channels a decade apart would enable us to handle a greater range of thicknesses.

We have found that wobble or surface unevenness of tubes can cause baseline fluctuation, and experimentation has shown that this is observable for steps as little as 0.0001 inch. This is not due to differences in air thickness, but rather to the geometric characteristics of the beta-ray collimator. This problem can be overcome, however, by cleaning deposit from narrow band spaced along the strip to provide a reference level of backscatter.

Although the beta-ray backscatter equipment has given reproducible results, a satisfactory method of calibration which relates backscatter values to the actual thickness of the deposit is still being pursued. For our purposes, such calibration, while intellectually satisfying, is not actually necessary as long as we compare deposits on tubes of the same composition. The output of the device is assumed to be proportional to the number of atoms of material on the tube under examination, if the average atomic number does not depart too seriously from that of carbon; this requires a low concentration of higher atomic number elements. This will generally be the case.

Calibration of the instrument thus far has been attempted with nitrocellulose films wrapped around an aluminum coker tube. These films have been separately cast and then taped to the tube for rating by the backscatter equipment. The film is then removed and the thickness of it measured independently by a microscopic light diffraction technique. Roughly, we have found by using this method that 1 inch on the strip chart corresponds to a thickness of 550 Å on an aluminum tube. However, the film so applied has always been wrinkled and nonuniform; hence, the search for an accurate method of calibrating the instrument is still underway, and an absolute calibration was not available in the earlier work. For that reason, deposit measurements are reported here in nondimensional units, but are approximately equal to thicknesses in Angstroms.

As a part of the overall calibration problem we needed a calibration method for deposits on different substrate metals. To implement this investigation, a calibration rod of 1/8-inch OD has been constructed which consists of sections of the following materials: brass, silver, aluminum, cadmium, cobalt, zinc, iron, tin, titanium, magnesium, carbon, and TEFLON<sup>®</sup> rod. By scanning this rod we obtain a series of steps on the recorder due to the different backscattering efficiencies of the materials of the rod. A 1000 Å PARYLENE<sup>®</sup> film then is wrapped onto the tube under tension, thus avoiding the previous wrinkling problem. The tube is then rescanned to obtain the calibrations for deposit thickness. An additional advantage of the multimaterial tube is that it will be available for restandardizing the tube when it becomes necessary to change windows, or to make regular calibration checks in the event of drift in the electronic equipment.

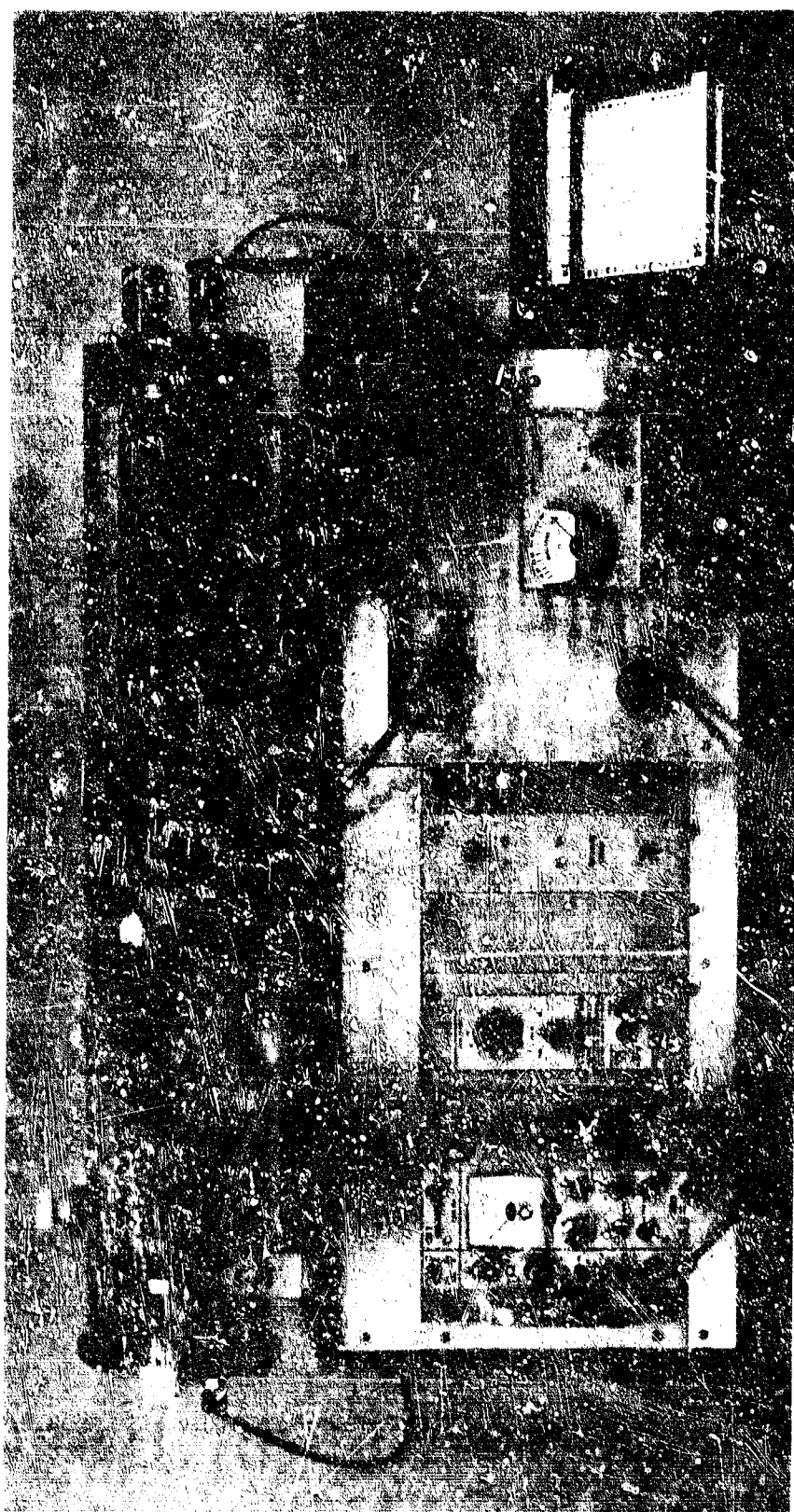


Figure 24. GEIA-RAY BACKSCATTER INSTRUMENT FOR MEASURING COKE DEPOSITS

Calibration with other pure hydrocarbon films is planned, although calibration with nitrocellulose is not considered to introduce a serious error, since the difference in the beta-ray backscattering by nitrogen and oxygen and the backscattering by carbon is not expected to introduce a significant error. However, this will be checked.

#### Boeing Titanium Strips

We have completed an evaluation of the tank deposit strips that were sent to us by Mr. George Hays of Boeing Aircraft Company. Results of these surveys are plotted as deposit profiles in Figures 25-28, where deposit thickness in  $\text{\AA}$  (approximate) are plotted versus distance along the strips. The narrow vertical dips shown are due to the fact that divider strips were welded and cemented to the flat titanium sheets, from which the fuel tanks were constructed, and were then removed. The greatest thickness of deposits was in the region of the divider strips, and this suggests a possible interaction of fuel with the cement or sealant, or an electrostatic attraction of deposit towards the dividers. The thickness of the sealant is indicated by the ends of the dips and ranged from an apparent 150 to 800  $\text{\AA}$ .

In order to compensate for the departure from flatness of the strips, the deposit was periodically removed by mild abrasive action, although this is not shown on the deposit profiles. By assuming that the minimum deposit thickness midway between divider strips represented the true unhindered depth, approximate dashed curves have been sketched in. It can be seen from the figures that apparent deposit thicknesses ranged from about 500 to 1400  $\text{\AA}$ . Calibration was made with a 1350  $\text{\AA}$  nitrocellulose film on cleaned titanium strips.

An effort was made to check these results by cutting two sections 2.5 cm in length with apparently uniform deposits from one of the strips. One of these areas measured 1200  $\text{\AA}$  and the other 1400  $\text{\AA}$  by beta-ray backscatter. These were then analyzed by combustion for carbon and hydrogen and a clean, freshly abraded sample was also analyzed for background impurities. Great care was taken to prepare these specimens so as to avoid contamination, and the edges and backs were filed clean with a fine file. The samples were then rinsed with normal heptane and dried in a vacuum oven for two hours at 150°C. The amount of carbon and hydrogen determined for the deposited samples, however, was much higher than would be expected from an oxidized hydrocarbon film of the thicknesses indicated by beta-ray backscatter, calculated densities being about 4.0 and 6.1 gm/cm<sup>3</sup>. This could have been the result of the inclusion of substantial amounts of oxides of heavy elements in the coating, since they would scatter more effectively than titanium, and would make the deposit appear thinner to the instrument. However, scanning the strips with the IES instrument (Varian Corporation) indicated that no large concentration of elements existed in the film other than carbon, hydrogen, and oxygen. At the present time the most reasonable explanation for this phenomenon is either that the original calibration of the instrument was at fault or that the deposit retained substantial amounts of the normal heptane with which it was washed, in spite of being dried in a vacuum oven for two hours at 160°C. This will be checked further.

Figures 25, 26, 27 and 28 follow

### Alcor JFTOT and SD/M-7 Coker Tubes

A few aluminum and stainless steel tubes used in our Alcor JFTOT unit have been examined both in the Tuberator and with the beta-ray backscatter device.

A distinct advantage of the beta-ray backscatter method is that it enables one to know when an observed color is not a true deposit, but rather the color of the metal itself. This was demonstrated with a stainless steel Alcor JFTOT tube having a faintly yellow tinted area which one might have rated a code 1-1/2. Nevertheless, the area still looked like bare metal, and indeed the beta-ray backscatter scan showed no difference between the tinted and the original metal colored areas. The beta-ray backscatter readout was constant for this entire tube.

However, a stainless steel Alcor JFTOT tube having an apparent code 2-1/2 rating, and giving the impression of being a true deposit overlay, gave a definite beta-ray backscatter count indicative of a deposit, as shown in Table 34. Here only the maximum visual code rating was taken, and then only on the side with the heaviest deposit. The beta-ray backscatter readout was, of course, on the entire tube, and shown in Table 34 is the range of thicknesses for both the heaviest and lightest deposits on opposite sides of the tube. The deposited area was about 1-1/4 inches long, but no effort was made to reduce the code ratings into shorter lengths, since most of the deposited area was the maximum code color, with lighter color around the fringes. This is rather typical of JFTOT deposit appearances.

Table 34. COMPARISON OF ASTM CODE AND BETA-RAY BACKSCATTER RATINGS FOR ALCOR JFTOT STAINLESS STEEL AND SD/M-7 COKER TUBES

Tube Designation	ASTM Code Tube Ratings												
	1	2	3	4	5	6	7	8	9	10	11	12	13
JFTOT Stainless Steel <sup>a)</sup>	( 2-1/2 )												
SD/M-7 Coker Run 525 <sup>b)</sup>	1	1	1		1/2	1/2	1/2	1/2	0	0	0	0	0
Thickness Range by Beta-Ray Backscatter, Å													
JFTOT Stainless Steel <sup>a)</sup>	( 140-700/480-340 )												
SD/M-7 Coker Run 525 <sup>b)</sup>	160-175	140-160	50-110	35-50	50	50	50	25	( no deposits )				

a) per cm.

b) per inch.

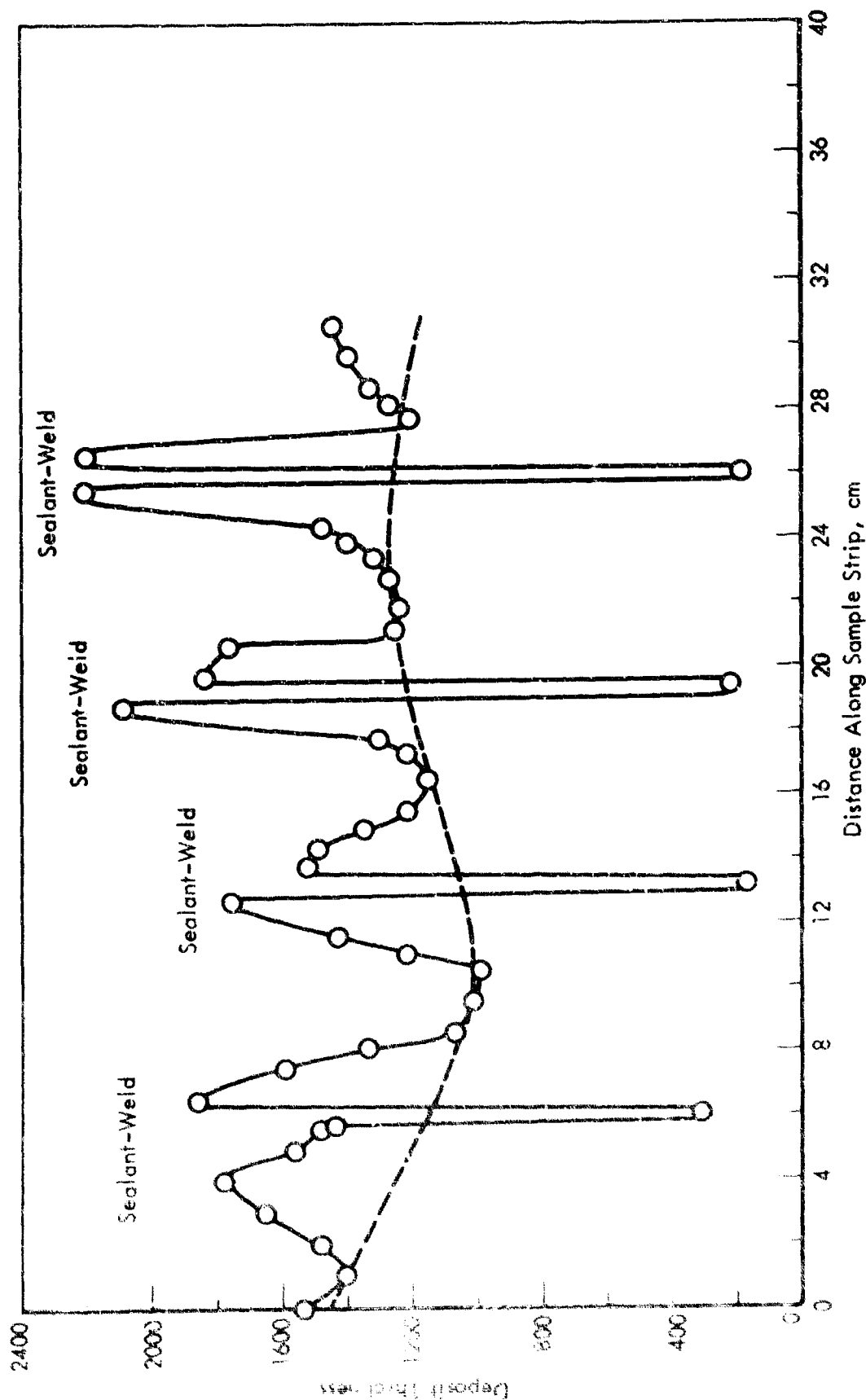


Figure 25. DEPOSIT PROFILE: SAMPLE STRIP NO. 2 FROM BOEING AIRCRAFT COMPANY

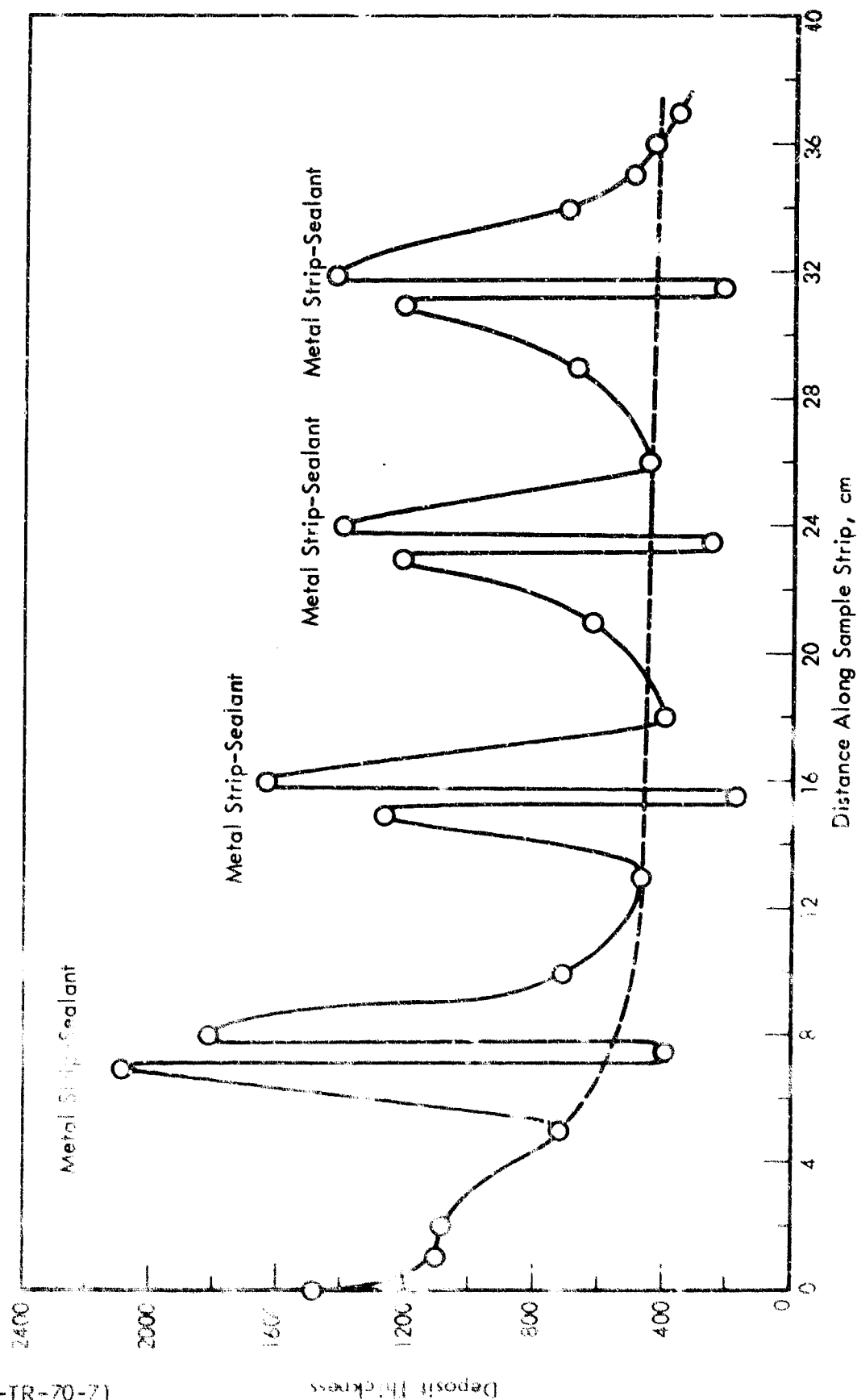


Figure 26. DEPOSIT PROFILE: SAMPLE STRIP NO. 3

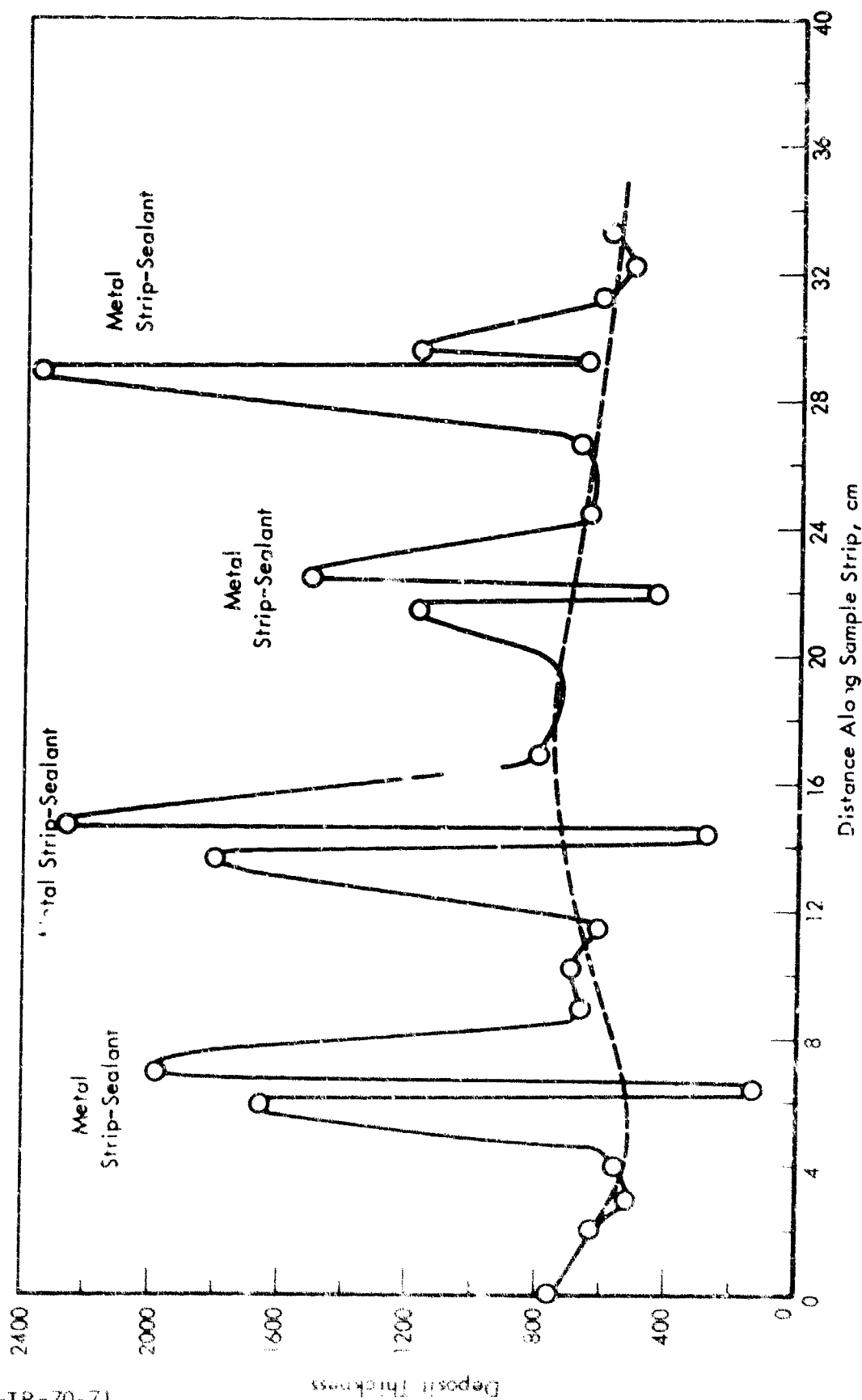


Figure 27. DEPOSIT PROFILE: SAMPLE STRIP NO. 4

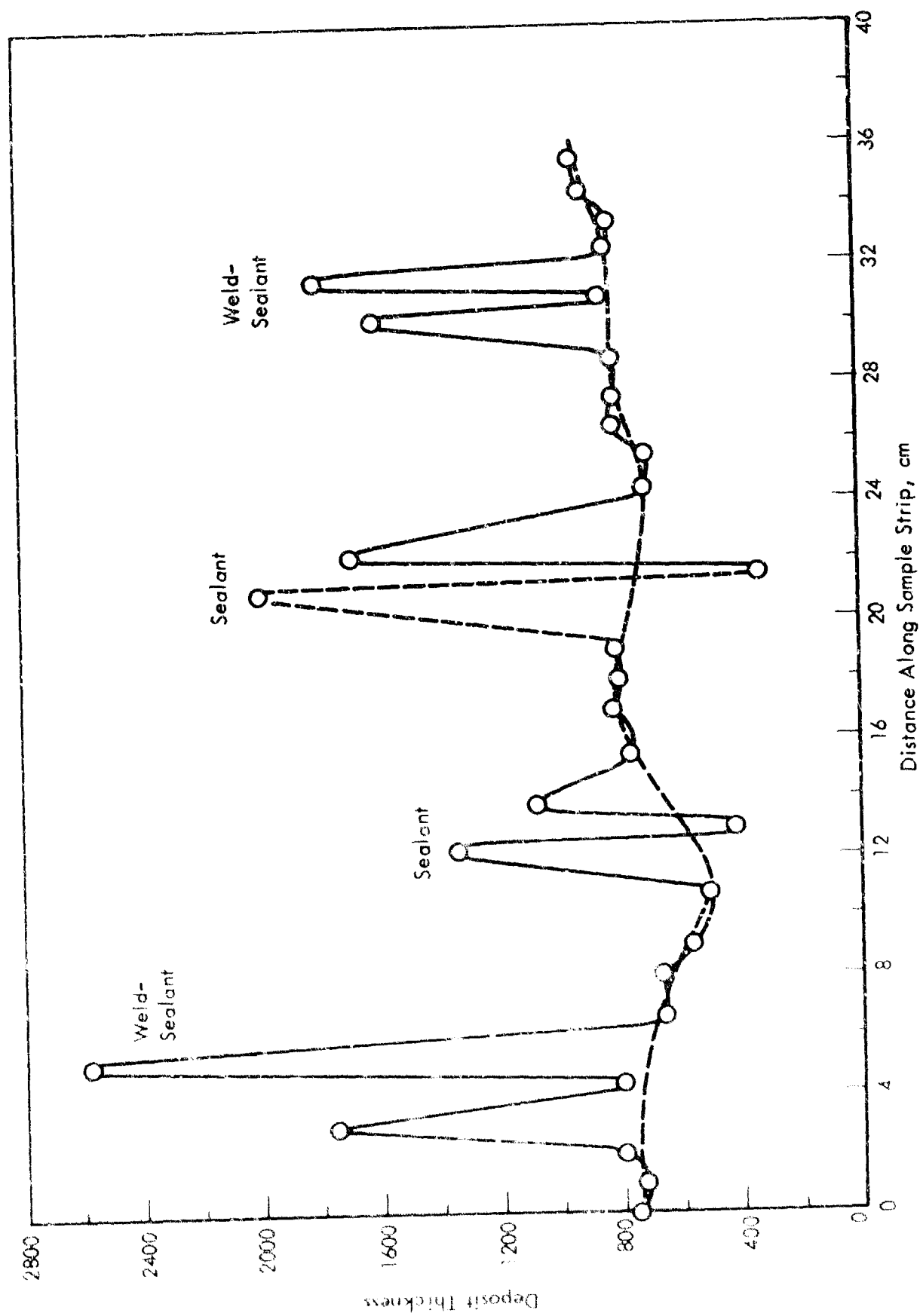


Figure 28. DEPOSIT PROFILE: SAMPLE STRIP NO. 5



One should not take the deposit thicknesses shown as being precise, since calibrations on stainless steel had not been made when this tube was measured. Moreover, since bare metal backscatter is a function of true metal composition, true calibration must be done on the same metal composition as that on which the deposit is measured.

The ASTM code versus the beta-ray backscatter ratings of a lightly deposited SD/M-7 outer tube deposit is also shown in Table 34. Actually, the areas shown do not correspond exactly, the area "1" for the backscatter ratings starting about an inch further away from the fuel outlet point than that of the ASTM code. One has to keep in mind this point in studying the other tube rating comparisons; the starting points are not necessarily the same. However, the regions in which deposits are located are in approximate agreement, the beta-backscatter results being much more discriminating than the visual code ratings.

The scanning width of the beta-ray backscatter instrument is slightly greater than  $1/8$  inch, since the radiation source slit is  $1/8$  inch wide and about  $1/8$  inch from the nearest point of the tube surface. The beta-ray backscatter readout is an average measurement of deposits for an area about  $1/8 \times 1/8$  inch. Actual deposit boundaries can be detected accurately, but the thickness at the boundary cannot be determined precisely because of the slit width. Alcor JFTOT tubes have a diameter of only  $1/8$  inch; hence, the backscatter measurement is influenced by tube diameter, or surface curvature. Consequently, for careful work calibrations would be necessary not only for every tube material, but for different tube diameters and for flat surfaces.

#### CRC Erdco JFTOT Preheater Tubes

A series of ten Erdco JFTOT preheater tubes from the CRC JFTOT evaluation program were supplied to us and rated by both the beta-ray backscatter method and our own laboratory ASTM Tuberator. The results of these ratings together with the average values of the ASTM code ratings from the panel evaluations and the results with the Erdco Reflectance Rater are tabulated in Table 35, and represented graphically (except for the values for the two most heavily fouled tubes) in Figures 29 and 30.

These tubes are  $5/8$ " OD and  $8-1/2$ " overall length. All ratings are given as the maximum values within one-inch long sections of the tube, numbering from the fuel outlet end. Actually the beta-ray backscatter readout was in the form of a sine wave, because the deposits were situated mainly on one side of each tube and the tube rotates and translates during the rating procedure. However, for the present comparison we have simply given the maximum rating or deposit thickness in A for each inch of the tube length. In the majority of cases, the geographical distribution of the deposits are found to be about the same by all methods of rating. That is, the deposits are heaviest near the exit end, although there are a couple of exceptions to this rule as shown with Tubes 10 and 2. Probably the two most interesting cases are tubes 7 and 15, which by both our own ratings and the CRC average fall in the critical region of code 2 to 3. Here the results with the beta-ray backscatter apparatus suggests that Tube 15 should fail whereas Tube 7 would probably pass, while the measurements with the Erdco Reflectance Rater would lead to the opposite conclusion. Although the Erdco Rater seems

Table 35. COMPARISON OF BETA RAY BACKSCATTER RATINGS OF CRC ERDCO  
JFTOT TUBES WITH THOSE FROM THE ASTM TUBERATOR  
AND THE ERDCO REFLECTANCE RATER

Tube Sections, Inches	Tuberator Ratings		FRDCO Rater	Beta Ray Backscatter, A
	CRC Ave.	Shell		
Tube No. 2, P. and W. No. 56 8 1/2" x 5/8" Tubes				
0-1	1.5	0.5	0.04	30
1-2	1	0	0.02	0
2-3	0.5	0	0.01	30
3-4	0.5	0	0.01	80
4-5	0.5	0	0.01	110
5-6	1.5	0.5	0.05	360
Tube No. 3, P. and W. No. 34 (8 1/2" x 5/8")				
1	2	3	0.08	820
2	3	5	0.17	1130
3	3	4.5	0.08	580
4	1	0.5	0.03	none
5	1	0.5	0.02	none
6	0.5	0.5	0.02	none
Tube No. 4, P. and W. No. 138				
1	2.5	3	0.15	1290
2	3	6.5	0.21	1790
3	3	6.5	0.21	1210
4	2.5	0	0.04	220
5	1	0	0.03	0
6	0.5	0	0.02	0
Tube No. 7, P. and W. No. 38				
1	2.5	1.5	0.16	680
2	2.5	1	0.11	440
3	1.5	0.5	0.08	220
4	1	0.5	0.04	110
5	1	0	0.02	30
6	0.5	0	0.02	0
Tube No. 10, P. and W. No. 80				
1	1	1.5	0.06	30
2	1.5	1.5	0.07	0
3	1	1.5	0.07	60
4	1	0.5	0.06	140
5	1	0	0.04	140
6	1	0	0.02	80

(Contd)

Table 35. (Contd). COMPARISON OF BETA RAY BACKSCATTER RATINGS OF  
CRC ERDCO JFTOT TUBES WITH THOSE FROM THE ASTM  
TUBERATOR AND THE ERDCO REFLECTANCE RATER

Tube Sections, Inches	Tuberator Ratings		ERDCO Rater	Beta Ray Backscatter, A
	CRC Ave.	Shell		
<u>Tube No. 12, P. and W. No. 90</u>				
1	2	0.5	0.08	580
2	1.5	0.5	0.07	190
3	1	0	0.03	0
4	1	0	0.03	0
5	0.5	0	0.02	0
6	0.5	0	0.02	0
<u>Tube No. 14, P. and W. No. 118</u>				
1	2.5	5	0.13	1400
2	2.5	4	0.14	1140
3	2	2	0.06	470
4	1	0.5	0.03	30
5	1	0	0.02	0
6	0.5	0	0.01	0
<u>Tube No. 15, P. and W. No. 137</u>				
1	3	2	0.11	1620
2	2.5	1.5	0.09	1260
3	2	1.5	0.06	910
4	1	1	0.04	600
5	1	0.5	0.03	360
6	0.5	0	0.02	220
<u>Tube No. 17, P. and W. No. 102</u>				
1	1	0	0.05	0
2	1	0	0.05	140
3	1	0	0.05	140
4	1	0.5	0.05	310
5	1	0.5	0.04	240
6	1	0.5	0.02	200
<u>Tube No. 18, P. and W. No. 9</u>				
1	1.5	1	0.08	880
2	3	3	0.16	1320
3	3	5.5	0.12	1700
4	1.5	0.5	0.02	630
5	1	0	0.01	80
6	1	0	0.01	0

to give quite consistent values within itself, the disagreement with the beta-ray backscatter apparatus is quite marked as shown in Figure 31. Although these data have not been analyzed statistically, the lack of concordance between the two methods of rating is evident. Of course, some of the scatter must be due to the beta-ray backscatter instrument, but we cannot assign this precisely since complete data on reproducibility will not be obtained until development work is complete. However, repeat runs on the same tube do not show deviations greater than about  $\pm 10$  A. The relation between ASTM Tubercator ratings and the beta-ray backscatter rating is shown in Figure 32 for both the CRC average ratings and the Shell ratings. Again a great scattering of results is noted. The CRC average ratings increase with a reduced slope for higher values because of the maximum 4 code rating imposed on those values.

A summary comparison of ratings by the other methods with the maximum deposit found with the beta-ray backscatter method is given in Table 36.

#### CRC Alcor JFTOT Preheater Tubes

We have also examined a group of ten tubes from the test program of CRC New Test Equipment Panel for the selection of a new thermal stability tester. Information received from the panel included ratings by an Alcor Mark 5 Tube Rater and Erdco Reflective Rater and values obtained by different panel members using the standard ASTM Tubercator. The latter values were averaged. The tubes were also rated on our own ASTM Tubercator equipment using our expanded scale (up to code 8), values being obtained every  $1/2$ " along the  $2-1/2$ " active length of the tube.

Values obtained by the various methods are given in Table 37 and are shown graphically in Figures 33 and 34, except for the two tubes with the heaviest deposits. Except for the Alcor Mark 5 Rater, which was obviously not sensitive enough, values obtained by the various methods showed general geographic agreement. The CRC Average ratings, of course, were always low in tubes having heavy deposits since the ASTM scale stops at 4. In general, the best agreement with the beta-ray backscatter method was exhibited by the Erdco rater (Figures 33A and 34A and D). However, in a number of cases (Figures 33B, C and D and 34 B and C) rather marked differences were observed. This is particularly true of tubes 2, 6, and 8 (Figures 33C and D, and Figure 34C), which would have presumably been failed by the Erdco rater but passed by the beta-ray backscatter instrument.

The relation between the various ratings and the beta-ray backscatter measurements are shown in Table 38 and Figures 35 and 36. The considerable scatter of the points is evident. However, in view of the favorable characteristics of the Erdco rater, it will be worthwhile to evaluate this instrument further using the beta-ray backscatter instrument as a standard.

With both the Erdco Reflective Rater and the beta-ray backscatter equipment, the absolute level of the values observed could very well be related to tube geometry. Certainly in the case of the backscattering principle, the calibration of the equipment with respect to actual film thicknesses was preliminary and subject to error in absolute accuracy.

Figures 29 through 32 follow

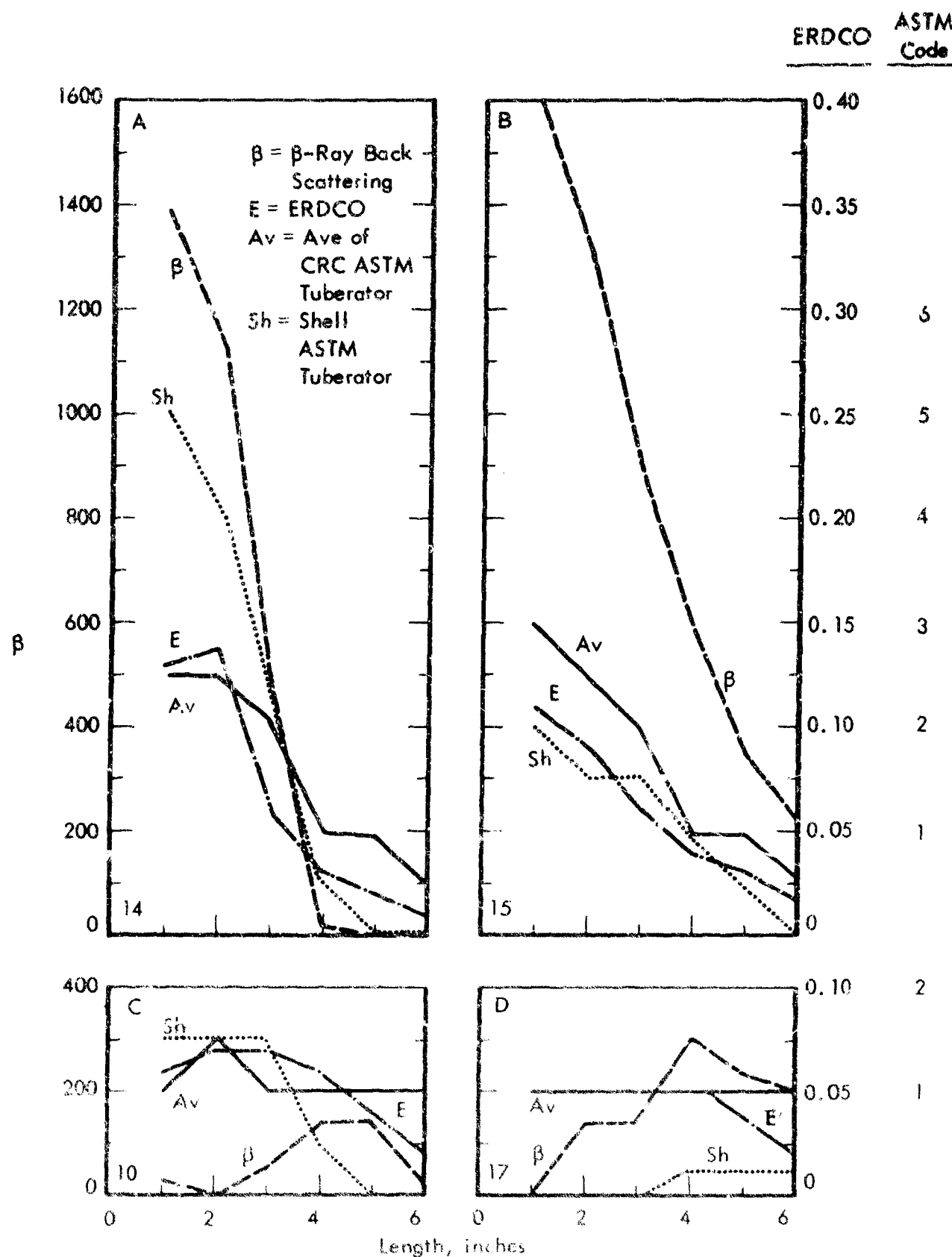


Figure 77. COMPARISON OF METHODS OF RATING ERDCO JFTOT TUBES

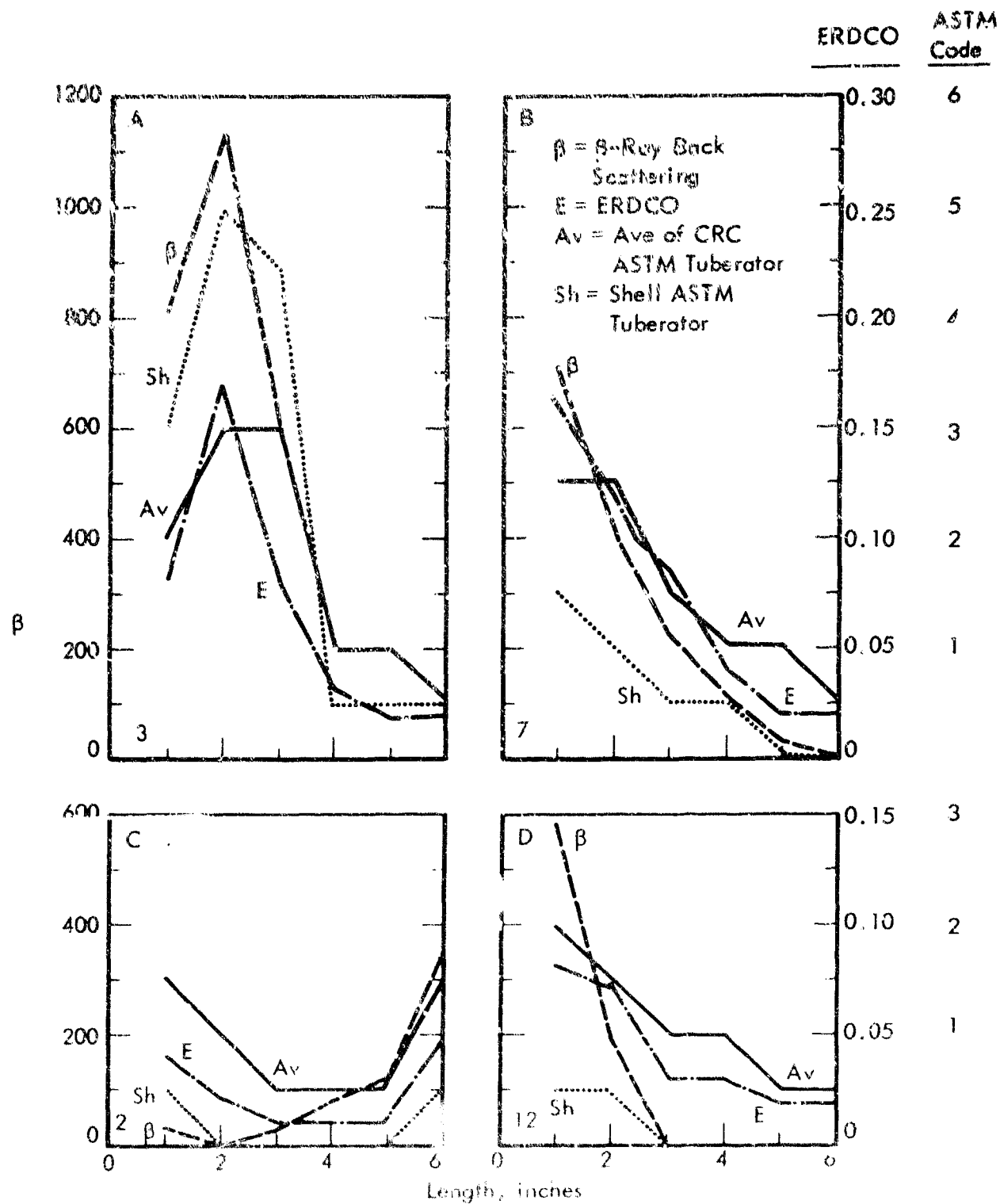


Figure 30. COMPARISON OF METHODS OF RATING ERDCO JFTOT TUBES

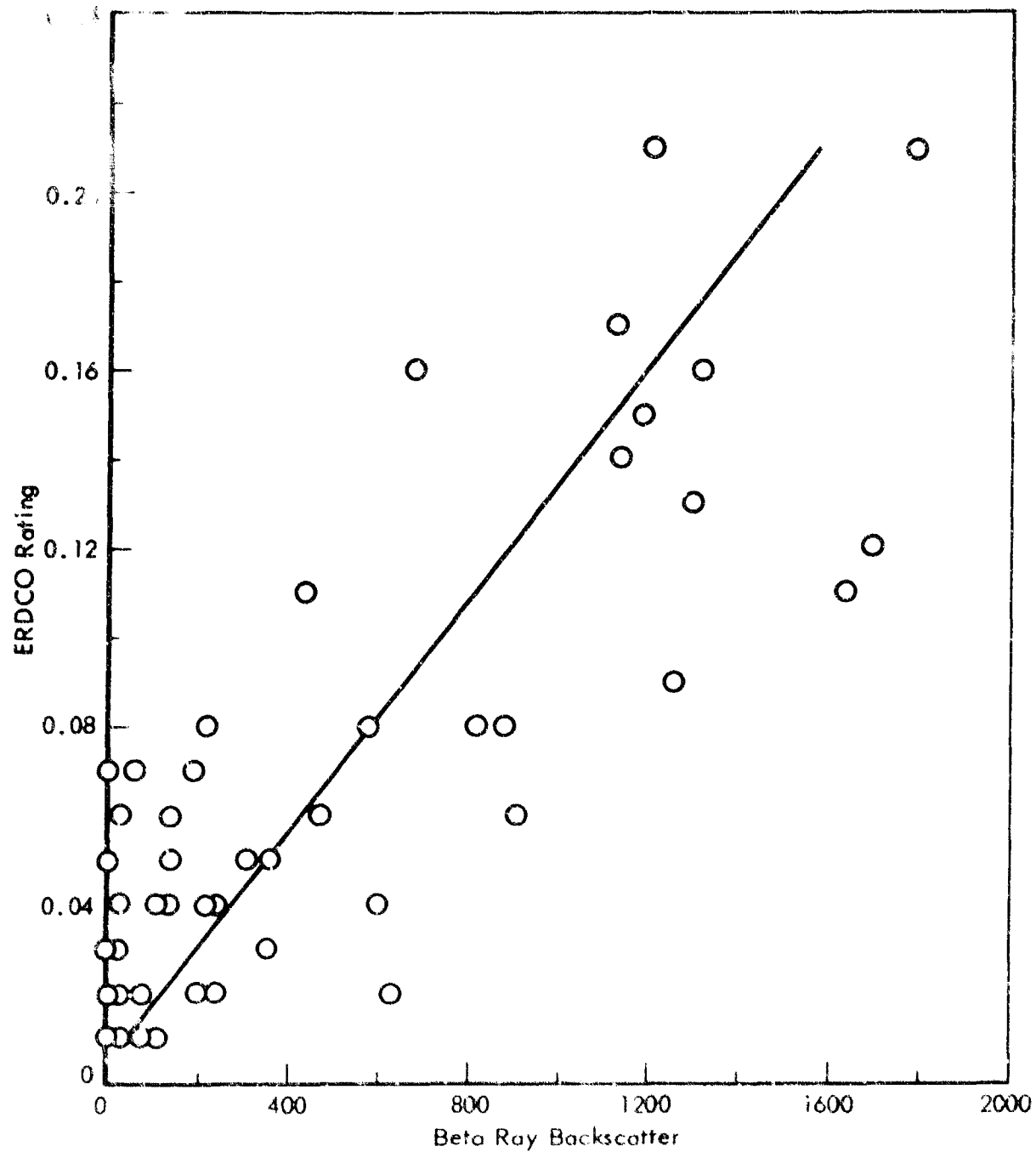


Figure 31. BETA-RAY BACKSCATTER RATINGS vs  
ERDCO REFLECTANCE RATINGS  
CRC Exchange Program ERDCO JFTOT Tubes

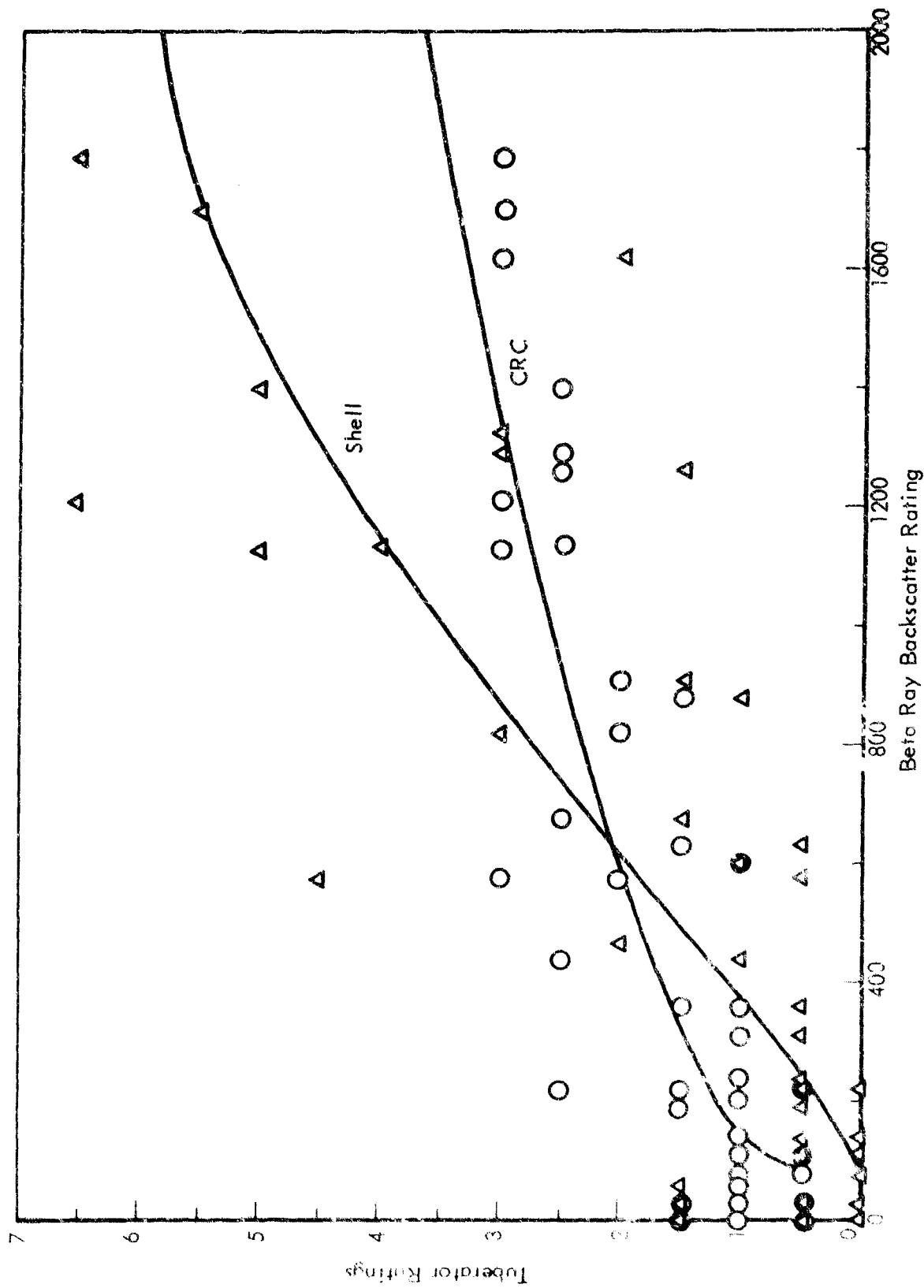


Figure 32. TUBERATOR RATINGS vs BETA-RAY BACKSCATTER RATINGS OF ERDCO JETOT TUBES  
 $8\frac{1}{2}'' \times \frac{5}{8}'' \text{ OD}$



Table 36. SUMMARY COMPARISON OF OTHER RATING METHODS  
WITH BETA-RAY BACKSCATTERING METHOD  
FOR C-1 ERDCO JFTOT TUBES

Tube No.	Max Beta-Ray	Rating of Beta-Ray Max by <sup>a)</sup>		
		ERDCO	Shell ASTM	CRC Ave.
4	1790	0.21	6.5	3
18	1700	0.12 (.6)	5.5	3
15	1620	0.11	2	3
14	1400	0.13 (.14)	5	1.5
3	1130	0.17	5	3
7	680	0.16	1.5	2.5
12	580	0.08	0.5	2
2	360	0.05	0.5	1.5
17	310	0.05	0.5	1
10	140	0.06 (.07)	0.5	1 (1.5)

a) Values in parentheses are maximum values according to specified method.

Table 37. COMPARISON OF BETA RAY BACKSCATTER RATINGS OF CRC  
ALCOR TUBES WITH THOSE FROM THE ASTM TUBERATOR  
AND THE ERDCO AND ALCOR MARK V RATERS

Tube Section, Inches	Tubulator Ratings		ALCOR MARK V	ERDCO Reflectance Rater	Beta Ray Backscatter, Å
	CRC Ave.	Shell			
Tube No. IIA1					
0-1/2	1.5	1	9.5	0.19	580
1/2-1	1.5	1/2	9.5	0.18	480
1-1 1/2	1.5	1/2	9.5	0.12	320
1 1/2-2	1.5	1	9.5	0.09	40
2-2 1/4	1	0	9.5	0.07	0
Tube No. IIA2					
1/2	1	1	9.5	0.15	200
1	1	1	9.5	0.15	120
1 1/2	1	1	9.5	0.10	40
2	1/2	0	9.5	0.08	40
2 1/4	1/2	0	9.5	0.06	40
Tube No. IIA4					
1/2	3	5.5	9.0	0.24	560
1	3	5.5	9.0	0.23	740
1 1/2	2	5.5	9.0	0.19	540
2	2	1	9.5	0.08	120
2 1/4	1	0	9.5	0.03	20
Tube No. IIA6					
1/2	3	2.5	9.0	0.15	95
1	3	3	9.0	0.18	140
1 1/2	2	1 1/2	9.0	0.12	40
2	1	1/2	9.5	0.04	50
2 1/4	1	0	9.5	0.03	10
Tube No. IIA8					
1/2	3	3.5	9.0	0.16	200
1	3.5	6	9.0	0.24	260
1 1/2	2.5	1	9.0	0.10	80
2	1	1	9.5	0.10	120
2 1/4	1	0	9.5	0.04	10

(Contd)

Table 37. (Contd). COMPARISON OF BETA RAY BACKSCATTER RATINGS OF  
CRC ALCOR JFTOT TUBES WITH THOSE FROM THE ASTM TUBERATOR  
AND THE ERDCO AND ALCOR MARK V RATERS

Tube Section, Inches	Tubulator Ratings		ALCOR MARK V	ERDCO Reflectance Rater	Beta Ray Backscatter, Å
	CRC Ave.	Shell			
Tube No. IIA14-13					
0-1/2	1	1/2	9.5	0.14	560
1	1.5	1/2	9.0	0.18	420
1 1/2	1.5	1	9.0	0.16	220
2	1.5	1/2	9.0	0.07	60
2 1/4	1	0	9.0	0.03	0
Tube No. IIA9					
1/2	4+	6.5	6.0	0.46	2480
1	3.5	7	5.5	0.60+	2480
1 1/2	1	1/2	9.5	0.06	0
2	1	1/2	9.5	0.03	0
2 1/4	1	0	9.5	0.01	0
Tube No. IIA10					
0-1/2	3	5.5	9.0	0.22	800
1	3	2.5	9.0	0.25	900
1 1/2	3	3	9.0	0.20	660
2	2	1/2	9.0	0.09	240
2 1/4	1	0	9.5	0.03	0
Tube No. IIA20-18					
0-1/2	3.5	3.5	9.0	0.21	680
1	3.5	3.5	9.0	0.22	800
1 1/2	3.5	3.5	9.0	0.15	480
2	2	2	9.5	0.06	80
2 1/4	1	0	9.5	0.02	0
Tube No. IIA22					
1/2	4.5	6.5	6.0	0.44	1860
1	4.5	7	5.5	0.60+	2460
1 1/2	1	1/2	9.0	0.12	80
2	1	1/2	9.5	0.10	0
2 1/4	1	0	9.5	0.07	0

Table 38. SUMMARY COMPARISON OF OTHER RATING METHODS  
WITH BETA-RAY BACKSCATTER METHOD  
FOR CRC ALCOR JFTOT TUBES

Tube No.	Max $\beta$ -ray	Rating at $\beta$ -Ray Max <sup>a)</sup>		
		Erdco	Shell ASTM	CRC ASTM Ave.
9	2480	0.60+	7 1/2	3
22	2460	0.60+	7	4
11A10	900	0.25	2 1/2 (5 1/2)	3
20-18	800	0.22	3 1/2	3 1/2
4	700	0.23 (0.24)	5 1/2	3
1	600	0.19	1	1 1/2
14-13	550	0.14 (0.18)	1/2 (1)	1 (1.6)
8	200	0.24	6	3.5
2	200	0.13 (0.15)	1	1
6	140	0.18	3	3

a) Values in parentheses are maximum values according to specified method.

Figures 55 through 56 follow

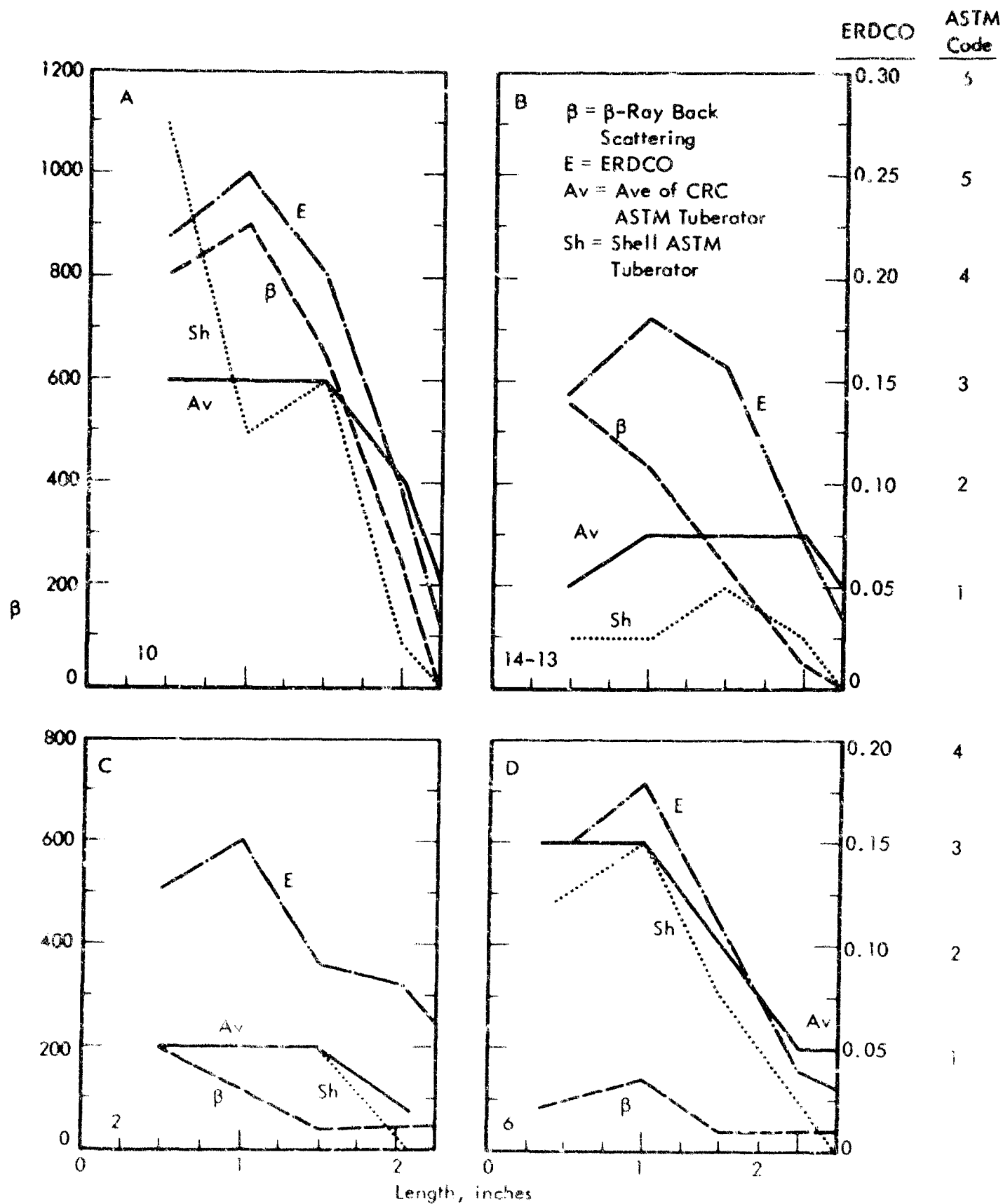


Figure 33. COMPARISON OF METHODS OF RATING ALCOR JETOT TUBES

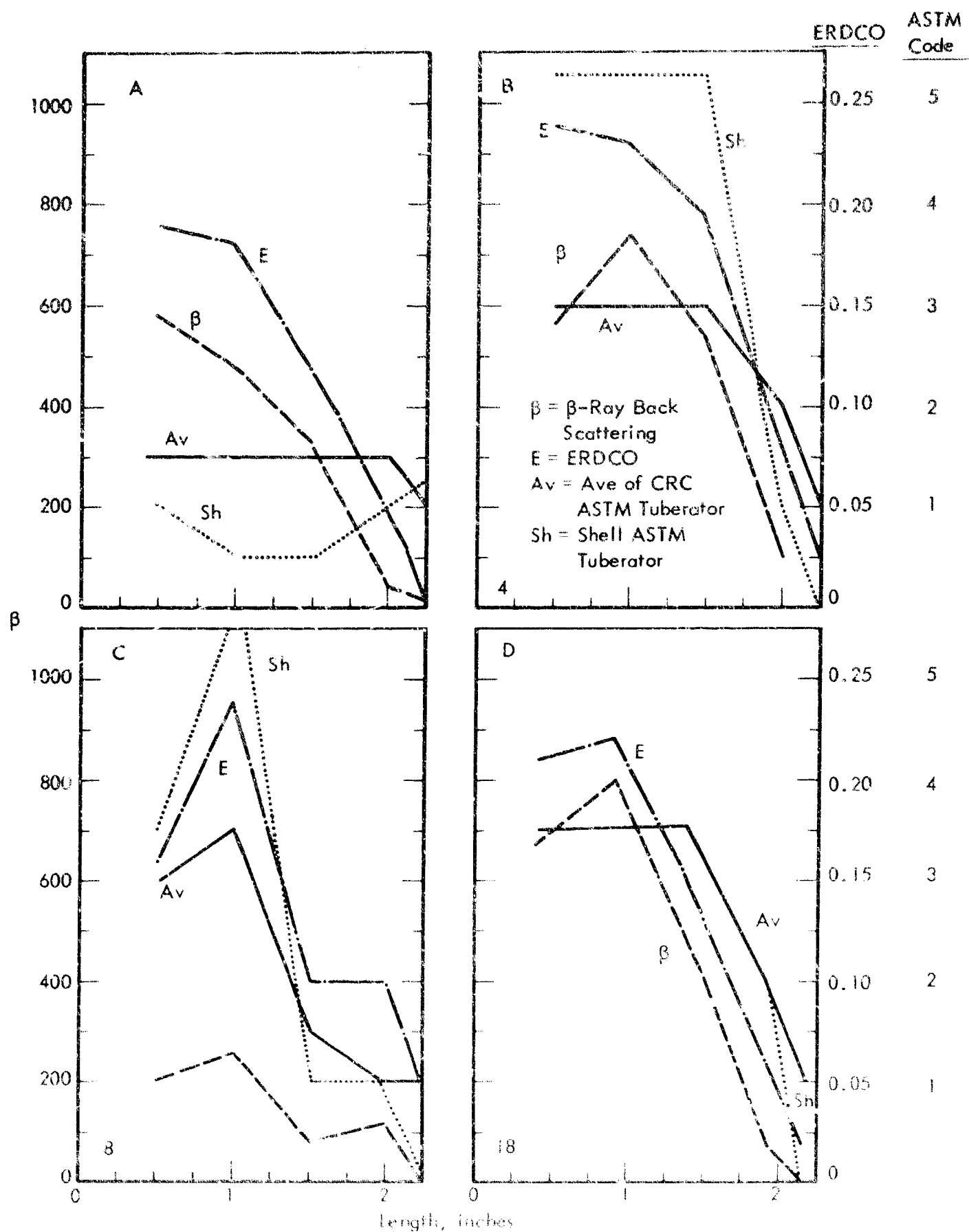


Figure 34. COMPARISON OF METHODS OF RATING ALCOR JETOT TUBES

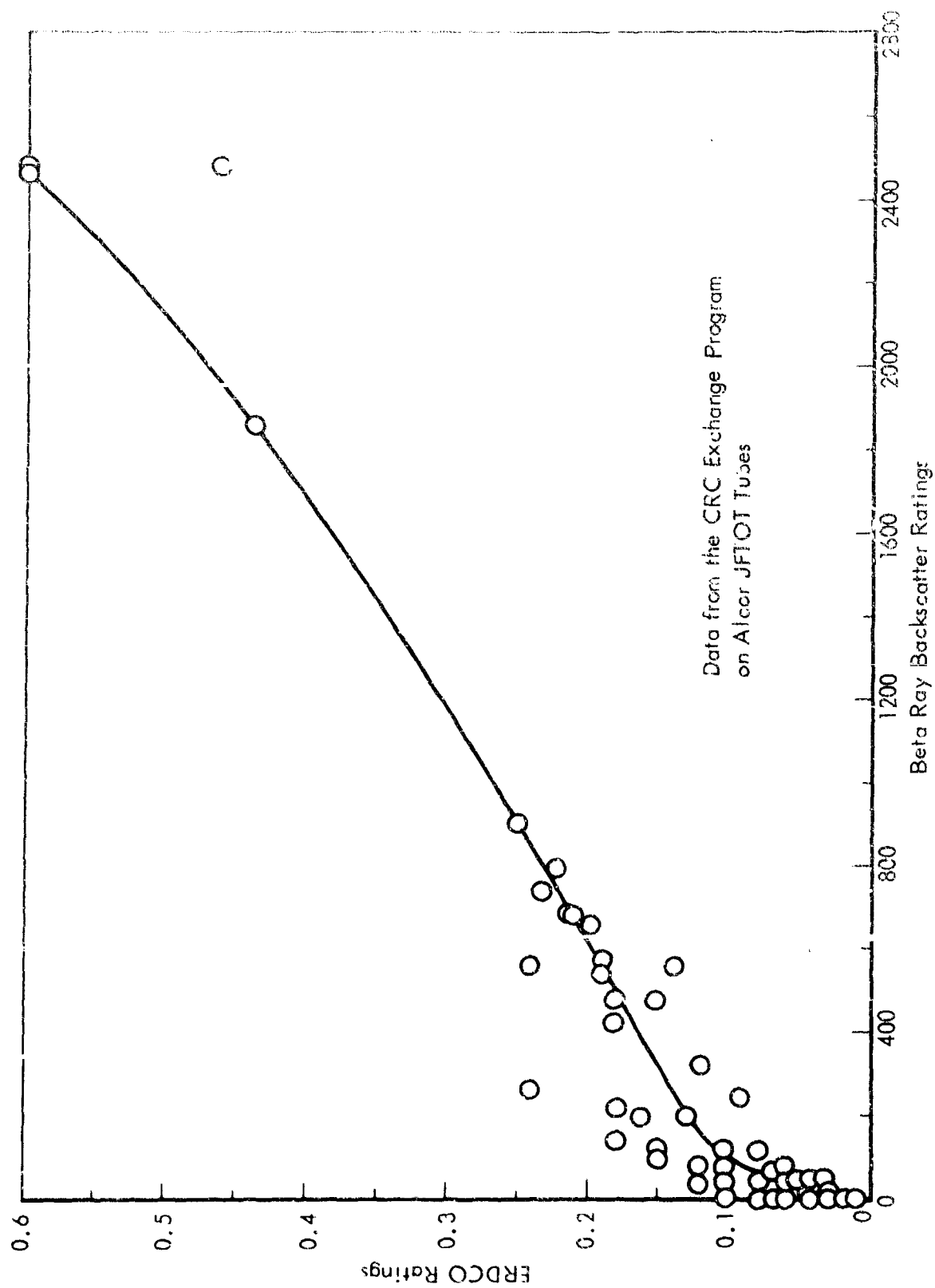


Figure 35 CORRELATION OF BETA-RAY BACKSCATTER RATINGS WITH THOSE FROM THE  
ERDCO REFLECTANCE RATER

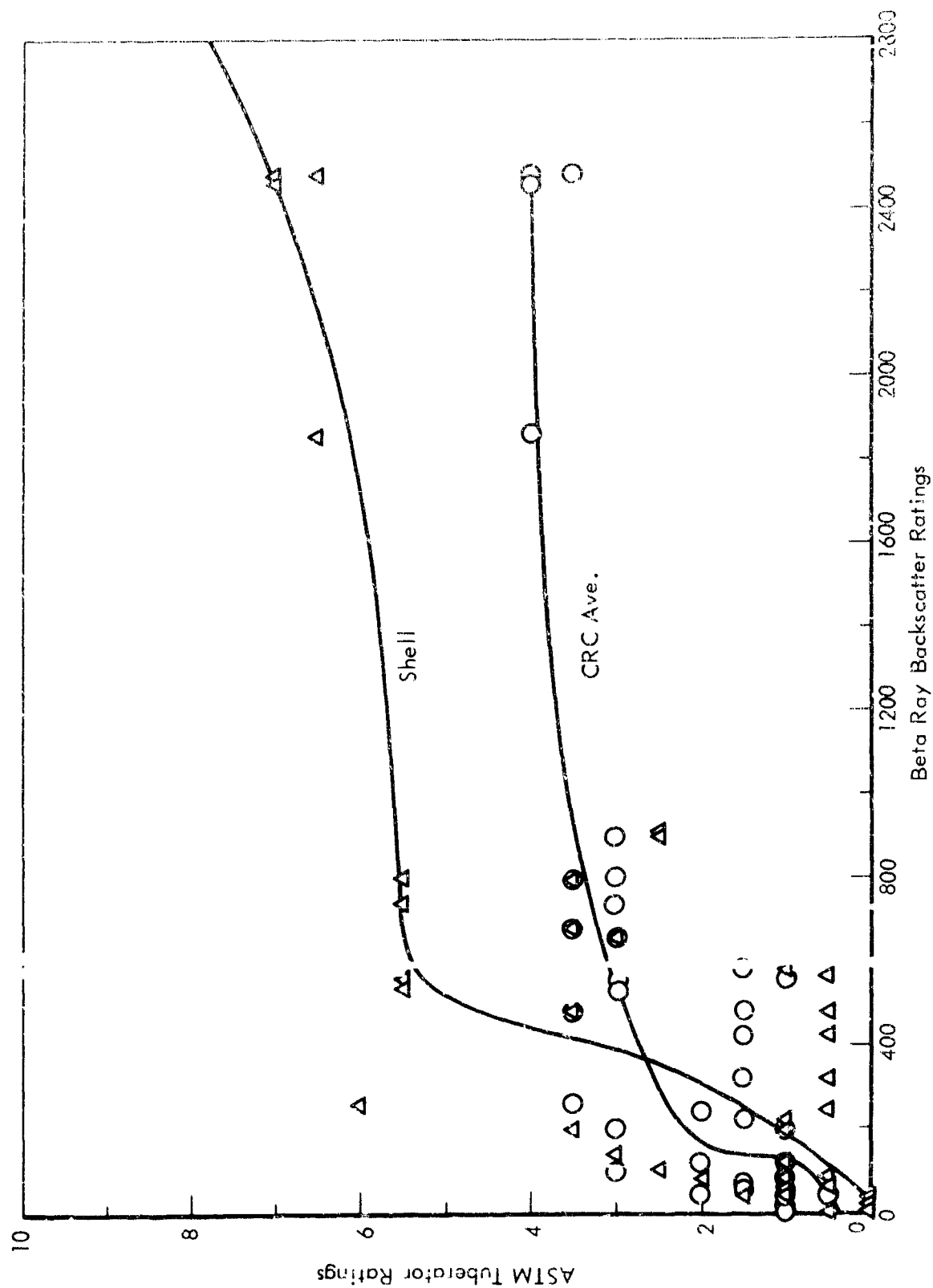


Figure 36. CORRELATION OF BETA-RAY BACKSCATTER RATINGS WITH ASTM TUBERATOR RATINGS OF  
ALCOR, FTOT, TUBES BY SHELL AND CRC EXCHANGE GROUP RATES



### Problems in the Use of The Beta-ray Backscatter Instrument

Although it is evident from the above that the beta-ray backscattering principle can be applied to the evaluation of the amount of deposit formed on a metallic surface, there are a number of problems which could complicate the application of the principle. One is the composition of the tube metal substrate. Since the intensity of the scattered electrons is roughly proportional to the atomic number of the element, it is obvious that a calibration must be established for each type of metal used on which deposits are measured. In addition, the composition of the deposit will have an effect on the apparent thickness. This is shown in the Table 39. However, it is evident that the amount of error introduced by the presence of the usual percentages of oxygen, sulfur, and nitrogen will not be large, and it is unlikely that any significant error will be introduced if an average composition of the deposit is assumed. The presence of large amounts of heavy metals or metal oxides in the deposit, however, would seriously hamper interpretation of the measurements. Another problem is the need for a standardized calibration procedure, which requires the production of standardized films with known thicknesses. So far we have been using nitrocellulose films, the thickness of which can be measured by optical refraction. Another factor which can influence accuracy is distortion of the surface or a serious wobble in a rotating tube. However, this has been tolerable at the level usually encountered with JFTOT tubes and warped plane surfaces by providing occasional clean surfaces on the specimen for measurement references. Another definite limitation is the thickness of the deposit measured. For each source there is a limit of thickness which can be measured as a result of saturation, i.e., capture of essentially all the electrons entering the deposit. Thus, the intensity of the source was chosen so that the instrument could measure the 2,500 Å thickness that we expected to encounter. Measurements of thicker deposits would require a more intense source.

Table 39. ESTIMATED ERRORS IN BETA-RAY BACKSCATTER  
MEASUREMENTS OF DEPOSIT THICKNESSES DUE TO  
NONCARBON ELEMENTS

Assumed Composition of Deposit	% non CH	Error In Deposit Thickness, %
CH	---a)	---a)
CH <sub>2</sub>	7, H	+2.0
CHO. <sub>25</sub>	23.5, O	-1.5
CHS. <sub>05</sub>	11.0, S	-3.5
CHFe. <sub>01</sub>	4.1, Fe	-2.5
CHPb. <sub>001</sub>	1.4, Pb	-3.1

a) CH was taken as the reference composition.

The present instrument is still under development, and it is anticipated that the above problems will be sufficiently resolved to allow the instrument to be used for evaluating the thickness of deposits on heat-exchange surfaces, as well as for other uses in which it is desired to measure the thickness of coatings.

#### Fuel Additives for Improvement of Thermal Stability

One important consideration in the development of fuels for high temperature operations is the decision as to what additives, if any, will be used with the fuels. Although the selection of the additives is best left until the conditions under which a fuel will be used are known, some preliminary investigation has to be done in order to ascertain the interaction between the fuel and different additives. One of the additives which is suspect is the metal deactivator, since it has a decomposition temperature of about 540°F in bulk.

Accordingly, a program has been initiated to screen potential additives for improving thermal stability, particularly of a variety which will function at higher temperatures. As a part of this investigation, we have taken a look at iodine, partly because of the lubricity improving characteristics of this material. It was reasoned that if iodine decreases the coefficient of friction, as has been reported under certain circumstances, then it might reduce the deposition tendency of fuels in a coker, where metal from pump wear might be a contributing factor. However, the addition of 0.1 percent iodine to PWA-535 jet fuel caused very severe deposit formation in the SD/M-7 coker at 600°F (code 5.5/56.5 compared to 2.5/16 for the iodine-free fuel). Consequently, no further testing is planned with this additive.

Following a lead from earlier work on the influence of metal environments on thermal stability, in which metallic zinc was thought to have a beneficial effect, zinc 2,4-pentanedione was tested in decalin at 600°F. The metal was added at a concentration of 200 ppm without effect (code 2.5/16.5 versus 2.5/15 for the nonadditive fuel) in the SD/M-7 Fuel Coker. Further observations with metallic zinc in decalin indicate that the benefit originally found was characteristic of the effect of metal deactivator (MDA; N,N'-disalicylidene-1,2-propanediamine) also present. MDA was found to provide improvements in thermal stability of decalin up to 600°F, but became ineffective at higher temperatures. This suggested the need for a chelating agent with a higher thermal stability, since MDA is not stable above about 540°F.

It had been concluded in the earlier tests that Zn did not interfere with the effect of MDA on the deposition tendency of decalin as did certain other metals such as Ni, Fe, Pb, and Cr. The results with zinc 2,4-pentanedione were therefore in harmony with this conclusion.

The beneficial action of MDA was assumed to be directly related to its action in passivating dissolved, and possibly surface metals, and not necessarily to the action of a zinc chelate which had been formed. Conceivably, even where concentrations of soluble metals are extremely low (ppb level), these metals may nevertheless play a key role in oxidative thermal degradation of fuels. Since virtually all fuel handling and storage equipment is metallic, metal is always present, and even in glassware tests of thermal stability the fuel has been previously in contact with metals.

It was concluded that perhaps a more stable chelating agent would extend the resistance of fuels to thermal oxidative degradation to higher temperatures than does MDA, even at air saturation conditions. Starting from this premise, some fifty compounds have been screened using the SD/M-7 fuel coker, of which six definitely have been found to be effective at 675-700°F. Probably several of the others would also prove effective at lower temperatures. A highly saturated jet fuel of a JP-7 type with high thermal stability (RAF-159-60, designated F-68) was used for the screening tests. This fuel has a thermal stability breakpoint by the SD/M-7 coker method of about 675°F. Other properties for this fuel are shown in Table 40.

The simplest member and first discovered of the new class of thermal stability additives (additive A) was found to improve the coker ratings of F-68 over the temperature range of 600 to 700°F. At the latter temperature the improvement in thermal stability is equivalent to ca 125°F. That is, the coker deposit rating at 700°F is equivalent to that obtained at 575°F without the additive.

Most of the other compounds tested have involved structural variations of the basic form of additive A by substitutions of groups and atomic species on and about the functional parts of the molecule. However, in the work done to date, no one simple mechanism appears to account for the activity of all the additives. Indeed, not all of the effective compounds are believed to be chelating agents. Some of the materials tested, although having some of the structural features of additive A, are entirely different in their chemical properties. Moreover, we have no simple explanation for the maximum temperatures at which these additives are effective. Structural modifications which increase the thermal stability of the additive do not necessarily improve its effectiveness. Most of these changes have resulted in less favorable activity, some decreasing the thermal stability of the blended fuel.

In four cases with the six effective additives, group substitutions at the functional group of additive A apparently did not harm the ability of additive A to enhance thermal stability, and in addition are expected to improve the resistance of the additive to water leaching. The fifth effective compound (additive A') is a different chemical type, but the structure was suggested by that of additive A. The sixth additive is a fluorinated form of additive A ( $A_F$ ). The effectiveness of these six compounds and MDA is shown in Table 41. Although the effectiveness of the additives increases with temperature, the results shown in Table 41 demonstrate how an additive may first be beneficial and then become harmful above some critical temperature. None of the additives has been found effective at 725°F, when tested at that level, and one additive (A') that was beneficial at 675° was very harmful at 700°F. The cause for this sudden change in activity with temperature rise is due either to a thermal stability limit of the additive itself, or to a change in the thermal degradation mechanisms with which the additives interfere. The fact that MDA was not effective above 600°F in either decalin or F-68 jet fuel suggests limitations in the thermal stability of this additive. The more thermally stable additive  $A_F$  was found to be no better and even worse than additive A.

Table 40. DESCRIPTION OF JET FUEL RAF-159-60

Properties	50.7	Water Tol, F-791, 3251, Interface Rating Sulfur, D1266, %w	1
Grav ASTM D287, °API	399	Mercaptan Sulfur, D1323, %w	0.0007
Distillation, ASTM D86, °F	403	Corrosion, Cu Strip, D130, 2 hr at 212°F	nil
I. E. P.	407	Aromatic Content, D1319, %v	1B
5%	410	Bromine No. D1159	0.8
10%	413	Naphthalenes, F791, 3704T, %v	0.7
20%	418	Smoke Point, D1322, mm	nil
30%	423	Luminometer No., D1740	35
40%	428	Existent Gum, D381, mg/dl	100
50%	436	16-hr Potential Gum, D873, mg/dl	1
60%	450	Net Heat of Combustion, Btu/lb	2
70%	471	ASTM D240 (Faar 2600 Modified)	
80%	496	NBS 5917	
90%	533	Hydrogen, D1018, %w	18,903
95%	1.5	Copper, mg/liter	18,886
E. P.	0.5	Nitrogen, ppm	14.9
Residue, %	164	Basic Nitrogen, ppm	< 0.02
Loss, %	-32	Peroxide No., D1563	< 0.3
Flash Point, TCC, D55, °F	30+	Water Separator Index, Severity 15	< 0.04
Freezing Point, D1477, °F		Light Transmission, % at 425 mμ	99
Color Saybolt, D15		Sediment, mg/gal, 0.45 micron	100
Viscosity, D445:		Average	4.6
at 100°F	1.81	Range, Eight Determinations	1.8-9.8
at 0°F	6.75	Other	
at -30°F	12.9	Paraffins, %v Mass Spec	80
Aniline Pt, °F	182	Naphthalenes, %v Mass Spec	19
Aniline-Gravity Constant	9227		

Table 41. EFFECTIVENESS OF ADDITIVES IN IMPROVING THE  
COKER RATINGS OF F-68 JET FUEL

Based on SD/M-7 Coker ratings at 250 psig, air saturated fuel.

Additive	Volume Percent	Increase in Temperature (°F) at Equal Tube Code Rating							
		Liquid Temp. (°F):	600	625	650	675	700	715	725
MDA	0.67		0	-	-	-	-40	-	-
A	0.2		25	50	75	100	125	-	-50
A <sub>1</sub>	0.25		-	-	-	-	125	-	-
A <sub>1</sub>	0.20		-	-	-	-	-	130	-
A <sub>2</sub>	0.20		-	-	-	-	125	-	-50
A <sub>3</sub>	0.20		-	-	-	90	-	-	-
A <sub>4</sub>	0.20		-	-	-	90	-	-	-
A'	0.20		-	-	-	100	-125	-	-
A <sub>F</sub>	0.20		-	-	-	-	-	-	-140

Table 42. EFFECT OF ADDITIVE A ON SD/M-7 COKER RATINGS OF METHYLCYCLOHEXANE

(Air saturated, 250 psig)

Additive	Percent	Temp., °F	ASTM Code, max/total
-	-	450	1.0/7.5
-	-	475	1.5/9, 1/8.5, 1/5
-	-	500	8/32.5
A	0.2 + 65ppm Ionol	475	5/39.5
A	0.2 (Ionol-free)	475	6/26
A	0.2 (Silica gel treated) <sup>a)</sup>	475	0/0
A	475 (Silica gel treated, then H <sub>2</sub> O equilibrated)	475	3/10.5
Copper <sup>b)</sup>	3130 ppm	425	1.0/9.5
Copper <sup>b)</sup>	2920 ppm	475	1.5/6.0

a) Silica gel treatment to remove dissolved water.

b) Copper added as copper undecylenate.

Of course, we are most interested in thermal stability additives for endothermic fuels, and have therefore extended our investigations with additive A to MCH. The breakpoint of air-saturated MCH is about 480°F on the SD/M-7 fuel coker and rather sharp. Some of the experimental runs with MCH are shown in Table 42. The first run with MCH containing 65 ppm Ionol oxidation inhibitor was disappointing, in that it increased deposit formation. To test whether this was due to the large concentration of Ionol, we next ran a test with Ionol-free MCH, but with essentially identical results (Table 42). We then passed MCH through a silica gel column and repeated the test. As shown, additive A gave a deposit-free test at 475°F, compared to a code 1 rating without the additive. Presumably, this improvement was due to dissolved water having been removed. To test this hypothesis we put equal amounts of MCH and water together in a bottle on a laboratory shaker for 10 minutes, then carefully decanted off the MCH and added 0.2 percent of additive A to it. This sample gave a 3/10.5 SD coker rating. Although this was not as bad as the untreated MCH, it certainly demonstrated the deleterious effect of water on the additive. However, other reasons may exist for the improved response of MCH treated with silica gel to additive A such as the removal of other polar dissolved substances, or resinous or metallic particles with the gel acting merely as a depth filter.

To test the hypothesis that additive A acts by effectively removing suspended metal from the fuel, copper in the form of copper undecylenate was added to MCH. However, even the rather large amount of about 3000 ppm had no harmful effects on the rating of MCH (Table 42). Similar additions of 300 and 3000 ppm copper undecylenate to decalin had no effect whatever on coker ratings at 600 and 650°F, and the effect of additive A was not significant at 600 and 650°F (Table 43).

Table 43. EFFECTS OF ADDITIVE A AND COPPER UNDECYLENATE  
ON THE THERMAL STABILITY OF DECALIN

Additive	Concentration	Temp., °F	SD Coker Tube Rating, Max/Total
--	--	650	1.5/6.5
A	0.2	600	1.5/6.5
A	0.2	650	1.0/10.5
A	0.2	700	5/11.5
Copper <sup>a)</sup>	300 ppm	650	1.5/8.5
Copper <sup>a)</sup>	300 ppm	650	1.5/9.5
Copper <sup>a)</sup>	3000 ppm	650	1.5/6.5

a) Copper Undecylenate.

This strongly suggests that the action of additive A has nothing to do with the presence of dissolved copper, since copper appears to have no harmful effect on the thermal stability of decalin or MCH. It also suggests that the action of additive A may not be related to the catalytic activity of dissolved metals at all, but rather to the activity of metal surfaces or to some other dissolved species. There is yet the possibility that iron or nickel, perhaps present from the MCH synthesis catalyst, may be involved. Further work is needed in this area, since the A-type additives seem to provide a tool for studying thermal stability mechanisms in addition to their possible practical applications.

Since copper addition had no effect on the thermal stability of either decalin or MCH, actual tests of additive A with added copper were not tried. Instead, it is planned to try this approach in a fuel such as F-68 in which there is a marked benefit of additive A for the thermal stability.

Since these tests had been made on the SD/M-7 Coker, which operated on the recycle mode, a question arose as to whether the result we obtained with additive A was merely an artifact of the test method or perhaps of the fuels, since both fuels tested in the SD Coker were virtually 100 percent saturated. We therefore used a commercial production turbine fuel (F-187) in the Alcor JFTOT Fuel Test. Here we found the same kind and magnitude of benefits, however, without any treatment of the fuel in advance. Results of these preliminary tests are shown in Table 44.

Both the maximum tube temperature and the fluid effluent temperature are shown. In fact, as would be expected, the maximum code ratings for the nonadditive runs correlate with the tube temperatures rather than with the liquid temperatures. With reference to liquid temperatures, 0.1 percent of additive A increases the thermal stability of F-187 by about 117°F, but the metal temperature, which is more significant, is extended by 160°F. As before, additive A<sub>P</sub> failed to give any improvement. We do not intend to screen any more compounds of the A-type at this time. Instead, the effectiveness and concentration effects of the six superior additives are being tested on the heat sink fuels from the current program and on a few pure hydrocarbon types. The improvement in the thermal stability of MCH by additive A after silica gel treatment suggests that we should look further at the effects of pre-purification treatments, and also together with antioxidants, anti-icants, corrosion inhibitors, and lubricity additives in selected cases. This will be combined with storage stability studies of the most promising combinations of additives and fuels. As soon as supplies of SHELLDYNE-H<sup>®</sup> fuel are available, work will be started to select the most desirable additives for this material.

Most of the future work will be done using the Alcor JFTOT Fuel Tester, so that realistic concentration requirements can be determined. The fuel makes over a hundred passes per test in the SD/M-7 coker but only a single pass through the hot test zone in the JFTOT. Hence, testing in the JFTOT may indicate a greater effectiveness for the additive and a higher temperature or lower concentration requirement for its use, especially if the additive is limited by its own thermal stability.

Table 44. EFFECT OF ADDITIVE A ON ALCOR JETOT FUEL  
TEST RATINGS OF F-187 TURBINE FUEL

Additive - Percent	Temperature, °F		ASTM Ratings, maximum code
	Max. Tube	Liquid Effluent	
A - 0.16	579	411	0
A - 0.1	605	437	0
A - 0.1	680	485	1
A - 0.1	728	505	1.5
A - 0.1	724	528	2
A <sub>F</sub> - 0.1	724	528	8
-- --	568	411	2
-- --	580	408	3
-- --	575	409	2.5



Research on additives for improving the thermal stability of fuels is continuing in several directions:

1. The relationship of additive concentration to temperature level will be investigated.
2. Suitability of additives of this type to various other endothermic fuels will be explored, together with some study of the effect of fuel hydrocarbon type composition.
3. Possible interaction problems with other jet fuel additives and common impurities will be examined.
4. Interaction effects with catalyst systems now under study will be investigated.
5. Possible deleterious side effects may be uncovered, particularly with respect to other fuel properties and storage stability.
6. Studies of the theory and mechanism of deposit formation as related to the action of A-type additives will be pursued.

#### Hydrocrackate Jet Fuels

Modern refinery processing has progressed to the point that certain processed hydrocarbon mixtures may be sufficiently high in naphthene content to be good endothermic fuel candidates. Accordingly, we have measured the thermal stability of hydrocrackate jet fuels.

Hydrocracking is generally a two-stage process which is designed to reduce the molecular weight of gas oil components under hydrogenating conditions to produce fractions which can be incorporated into gasoline and jet fuels. This process is coming into increasing use, and will be relied upon more and more to supply the increasing demand for jet fuels in the future. Since hydrogen requirements and operating conditions are directly related to the amount of aromatics left in the hydrocrackate, economics demands the highest permissible aromatic content in the jet fuel boiling range. This could have an adverse effect on thermal stability. On the other hand, deep hydrocracking might produce a very stable fuel. Since there are no published data available on this subject, we have obtained two samples from the Shell Emeryville pilot plant operation, one from each stage, having 30 and 10 percent aromatics contents (designated F-155 and F-156, respectively). The former could be used for blending purposes with a predominantly paraffinic straightrun material, and the second might be used as fuel directly.

A series of SD/M-7 Coker runs on these two samples are shown in Table 45, from which code 2.5 tube rating break points of 405 and 355°F were estimated; the high aromatics sample from the first stage (F-155) was the more stable fuel, which was a surprising result. Both ratings are better than specifications for jet fuels; the 405°F breakpoint is characteristic of a good jet fuel. Filter plugging tendencies were actually more limiting than tube deposits, reaching values of 13 inches of mercury for pressure drops at 350°F for both samples. No fuel additives were included, and since these are samples from a rather small pilot plant they are merely suggestive of what would be produced from commercial scale plants. No further work in this area is planned at this time.

Table 45. SD/M-7 COKER RATINGS OF HYDROCRACKATE JET FUELS

Fuel	Temperature, °F	Filter Pressure Drop, <sup>1)</sup> psi	ASTM Code Ratings <sup>1)</sup> max./total
F-155	500	40	8/35
F-155	425	12.4	5.5/18.5
F-155	425	74.4	5/20
F-155	425	100	4/18
F-155	400	--	(2.5/8)
F-155	375	24.1	0.5/9.5
F-155	350	7.7	0/10
F-155	325	0.5	1/6
F-155	405	--	(2.5/10.5)
F-156	425	35.4	8/35
F-156	400	53.7	4/14
F-156	350	5.5	1/4
F-156	325	1.6	2/8
F-156	355	(6.4)	(2.5/10)

1) Bracketed values are interpolations to obtain breakpoint temperatures.

### Modifications and Assembly of Thermal Stability Apparatus

The ancillary equipment for the special high severity "barebones" JFTOT received from the Alcor Corporation has now been installed, including a constant speed pump, watt meter, and pressure transducer. The constant speed pump consists of a Zenith No. 1/2 pump driven by a 1/50 h.p. Bodine motor. However, this motor is slightly underpowered and we have now installed a Minarik Electric speed and torque controller, which has overcome this problem. The pump normally turns at 10 rpm, but is capable of flows four times that rate.

Temperature is controlled at the maximum tube temperature point, which is 0.85 inch from the discharge end for aluminum tubes and 0.6 inch for stainless and ordinary steel. A Guardsman West Controller is being used satisfactorily in this application. We have also installed a liquid effluent thermocouple in the discharge hole of the preheater with readout on a Honeywell strip recorder.

Nitrogen pressure on the system is read on a Heise Gage (0-1,000 psig), while the filter pressure drop is measured by a 0-5 psi Stratham Pressure Transducer and read out on another Honeywell strip recorder. Nitrogen pressure is controlled by a simple two-stage Victor regulator.

Voltage to the pump motor and preheater Variac is supplied by a Stabiline Automatic Voltage Regulator capable of controlling to  $\pm 1/20$  volt. All other equipment is the same as supplied by Alcor Inc.

Besides the standard aluminum tubes, tubes made of stainless steel and No. 1015 steel already have been obtained and tested. The steel tubes all appear to give more severe ratings than does aluminum, but several factors complicate this comparison, such as temperature profile and metal color change, so that true comparison awaits the proper application of the beta-ray backscatter deposit analyzer. Other metals such as Inconel, nickel, and Incoloy are also being obtained for comparison studies in JFTOT tubes. A complete list of tube metals now on hand as JFTOT tubes is shown in Table 46. Except for Haynes 25, these and aluminum have been run on a Jet-A type fuel for evaluation of catalytic effects of metals on fuel deposition tendency. The tubes are currently being rated by the beta-ray backscatter technique, and will be rated afterwards by combustion for comparison. Calibrations on the various metals are still being made using 1000 A nitrocellulose film. Following satisfactory completion of this work, the effect of these different tube metals will be determined on MCH.

Assistance was obtained from Alcor Inc. and from Pratt and Whitney, East Hartford, Connecticut, on obtaining some of these special materials. In general, tubing with a 3/16" OD and approximately 0.68" ID for fabricating these tubes is difficult to locate. Alcor Inc. has manufactured coker tubes by attaching 3/16" end pieces of gold-plated brass to the desired 1/8" test section, and we have obtained two of these tubes for testing. We are not sure whether the brass, although gold-plated, is entirely innocuous in the deposition mechanism.

The high temperature version of the JFTOT which we obtained was designed for operation up to 1000°F and 1000 psig. So far we have only operated the rig at 300 psig, and the highest tube temperature we have tested has been 724°F (528°F liquid effluent temperature). At this temperature and even lower, the tube becomes

Table 46. COMPOSITION OF TUBE METALS FOR JFTOT

Tube Metal	Typical Composition, %											
Metals in Stock	Ni	Co	Cr	Mo	Fe	C	Al	Cu	Mn	W	Si	Other
Hastelloy Alloy C <sup>1)</sup>	54.6	2.5	15.5	16	5	0.08	--	--	1.0	4	1.0	0.35 V
Seamless Monel 400 <sup>2)</sup>	65.17 <sup>3)</sup>		--	--	1.2	0.13	--	32.33	1.05	--	0.09	0.008 S
Inconel Alloy 600	72.03		15.5	--	10	0.15	--	0.5	1.0	--	0.5	0.015 S
316 Stainless	13.28	--	16.75	2.50	65.14	0.05	--	--	1.73	--	0.51	0.035 P + S
304 Stainless <sup>1)</sup>	9	--	19	--	72	0.08	--	--	--	--	--	--
446 Stainless	--	--	25	--	75	0.35	--	--	--	--	--	--
Nickel 200 <sup>2)</sup>	99.56 <sup>3)</sup>		--	--	0.04	0.06	--	0.01	0.26	--	0.04	0.005 S
Cartridge Brass	30	--	--	--	--	--	--	70	--	--	--	--
1015 Steel	--	--	--	--	99.85	0.15	--	--	--	--	--	--
TD Nickel <sup>1)</sup>	2 percent by volume Thorium Oxide dispersed in Ni											
L-605 (Haynes 25)	9.90	51.75	19.85	--	1.60	0.12	--	--	1.65	15	0.60	0.015 P + S

1) These tubes are also on hand in aluminized form.

2) Compositions for these alloys are measured values.

3) These values are the combined nickel-cobalt compositions.

bent during the run so that it is no longer concentric with the outer shell. Of course, this is due to the thermal expansion of the tube, which is restricted by the clamping restraints of the end seals. This event seems to cause an actual change in the temperature difference between the tube control point and the liquid effluent during the course of the run.

We have attempted to overcome this problem by substituting the rubber O-ring seals with TEFLON® seals, which are more slippery, and applying a high electrically conductive lubricant to the electrical end clamps, in the hopes that the inner tube might slip during expansion. However, to provide good electrical connection with the aluminum tubes the clamping must still be too tight to allow for slippage during thermal expansion, and the tubes still bend. Perhaps with steel tubes this approach may work, but some redesign might be required. The bending can be particularly bothersome with the beta-ray backscatter device since it alters the distance from the source to the tube. This causes a sinusoidal fluctuation in the recorder output even on bare metal and if extreme would preclude the use of this method.

Several comparisons have been made between the JFTOT and ASTM methods on the same commercial turbine fuels, as shown in Table 47. These are all production fuels of recent date. The two methods are within 1/2 code number of each other generally, and at least as close as the probable repeatability of either method.

Two runs on the same F-187 fuel using stainless steel JFTOT tubes are also included for comparison. However, until these tubes can be rated by the beta-ray backscatter method, the true relative effects of aluminum and stainless steel will not be known, since the steels themselves change color on elevated heating. We do know from preliminary tests that a code 2 1/2 rating on a stainless steel tube was found by beta-ray backscatter measurement to have a thickness of less than 50 Å, while a code 1/2 deposit on aluminum with a whitish appearance (normally thought to be thinner) could be readily observed.

Recent experience with the Alcor JFTOT Fuel Tester showed that we were experiencing errors in control temperatures due to worn insulation on the control thermocouple. This emphasizes the need to inspect carefully and regularly the condition of the insulation after each run and to replace the thermocouple as needed.

To improve the accuracy of our tube metal temperatures, we have installed a double thermocouple, one which actuates the West temperature controller and one which reads out on a millivolt strip chart recorder. The latter is much more accurate and readable than the West controller dial and is not sensitive to thermocouple resistance. Liquid temperatures are also read out on a strip chart recorder as before. We believe the millivolt strip chart recorder is a more accurate instrument for the measurement of temperatures than the digital Honeywell recorder used as standard equipment with the Alcor Fuel Tester.

#### SD/M-7 Fuel Coker

A Beckman Oxygen Analyzer (Model 778) has been installed on the SD/M-7 Fuel Coker. However, this instrument is limited to operation at or below 50 psi maximum pressure, and hence cannot be used continuously throughout the test period.

Table 47. COMPARISON OF RATINGS BY THE JFTOT AND ASTM METHODS

Fuel	Fluid Temperature, °F	Test Method	Max. Visual Code Ratings
F-185	418	JFTOT	4
F-185	417	ASTM	4
F-185	420	ASTM	4
F-187	420	JFTOT	3
F-187	411	JFTOT	2
F-187	411	JFTOT	3
F-187	409	JFTOT	2.5
F-187	408	JFTOT	3
F-187	420	ASTM	3.5
F-187	415	ASTM	3.5
F-187	415	ASTM	1.5
F-187	410	ASTM	1.5
F-187	410	ASTM	0
F-187	410	JFTOT (stainless)	6
F-187	411	JFTOT (stainless)	4

Since the test method depends upon diffusion of oxygen through a semipermeable membrane, the flow velocity of the test fuel past the detector head must be maintained at a minimum of 1.8 ft/sec. To accomplish this a special fitting with a restricted passage was designed and built. The analyzer actually measures the oxygen partial pressure, and therefore the pressure at the pickup must be known to permit calculation of  $O_2$  in ppmw. Static pressure at the pickup is being measured with a Statham pressure transducer readout system. Pressure drop across the restricted passage has been determined to be about 0.4 psi, and so the pressure at the pickup is simply taken to be 0.2 psi less than the upstream pressure reading. This is a trivial correction.

Currently the oxygen can only be measured at the beginning and end of the test, that is, before and after operating at high pressure. After measurement of  $O_2$  concentration, the detector, which is located on a by-pass leg at the discharge end of the pump, is isolated from the rest of the system for the remainder of the coker run. This is an undesirable condition, but it will suffice until a detector is designed and built which will withstand greater pressures.

#### STORAGE STABILITY OF MCH AND SHELLDYNE-H<sup>®</sup> FUELS

Storage stability tests which were started over four years ago have been completed and the results evaluated. At that time samples of pure MCH were stored in the 130°F hot room. Samples were packaged in one gallon epoxy lined pails with a 2:1 ullage ratio with oxygen concentrations from pure  $O_2$  to pure  $N_2$ . Several phenolic and diamine type oxidation inhibitors were included, as well as MDA, in some of the samples, while control samples contained no additives at all.

Examination of the samples for soluble and insoluble gum gave no indication of deterioration in any of the samples during this period of storage. No insolubles were observed in any case and steam jet gums were less than 1 milligram per deciliter. No indication of discoloration was observed. Finally, coker tests showed no loss whatever of thermal stability. In fact, the actual ratings were better than those obtained on the fresh material prior to this severe exposure.

The storage stability of SHELLDYNE-H<sup>®</sup> fuel over a shorter period has also been investigated in accelerated tests. Three different batches of SHELLDYNE-H<sup>®</sup> fuel with minor differences in bromine number have been exposed to 200°F constant temperature for 18 and 30 hours. Three different oxidation inhibitors were tested in comparison with the nonadditive material, and all samples were blanketed with pure oxygen. Exposed samples were examined for both soluble and insoluble gums.

Unfortunately, results of these tests showed no consistency, either with respect to exposure time or to the presence of inhibitors (Table 48). Apparently an unknown factor was influencing the results; the 450°F temperature of the standard steam jet was probably too low to drive off all the SHELLDYNE-H<sup>®</sup> fuel. Accordingly, the steam jet temperature was raised to 500°F and the results were more consistent. However, even on this basis the results are quite erratic and it is possible that some other factor is affecting the results.

On the basis of our present results, it appears that the phenolic inhibitor 2446B (duPont 22) is the most efficacious antioxidant for this fuel.

Table 48 follows

Table 48. STORAGE STABILITY OF SHELLDYNE-H® FUEL

Oven Tests at 200°F Under O<sub>2</sub> Blanket

Shelldyne-H® Sample Designation	0 - Hours			18 - Hours				30 - Hours			
	Insolubles ex Solvent mg/dl	Solubles ex Filtered Fuel 450°F mg/dl	Total Gum, mg/dl	Insolubles ex Solvent mg/dl	Solubles ex Filtered Fuel, mg/dl		Total Gum, mg/dl	Insolubles ex Solvent mg/dl	Solubles ex Filtered Fuel, mg/dl		Total Gum, mg/dl (500°F)
					450°F	500°F			450°F	500°F	
F-151 Isanol® duPont 22 duPont 30 —	0.4	14.6 <sup>a</sup>	15.0 <sup>a</sup>	0.0	3.2	5.4	5.4	0.0	16.2 <sup>a</sup>	6.4	6.4
	0.8	5.8	6.6	1.3	5.8	4.2	5.5	0.0	5.5	3.0	3.0
	0.6	6.0	6.6	0.0	16.6 <sup>a</sup>	3.8	3.8	0.4	9.2	5.6	6.0
	0.2	20.8 <sup>a</sup>	21.0 <sup>a</sup>	0.6	15.2 <sup>a</sup>	6.0	6.6	0.8	10.6	11.4	12.2
F-158 Isanol duPont 22 duPont 30 —	0.4	13.2 <sup>a</sup>	13.6 <sup>a</sup>	5.8	12.2 <sup>a</sup>	—	18.0	0.6	—	—	—
	0.4	10.4	10.8	2.4 <sup>a</sup>	2.2	—	4.6	0.0	5.0	0.0	0.0
	0.0	9.4	9.4	13.3 <sup>a</sup>	8.2	—	21.5	0.0	0.8	0.2	0.2
	5.2	0.2	5.4	0.0	7.8	—	7.8	0.2	7.0	7.0	7.2
F-157 Isanol duPont 22 duPont 30 —	0.4	1.0	1.4	0.0	6.4	—	6.4	1.2	5.2	3.0	4.2
	0.6	2.4	3.0	5.6	10.4	1.0	14.1	0.4	1.0	2.4	2.8
	0.2	2.6	2.8	9.3	4.8	—	13.7	0.4	5.4	—	5.8
	0.2	1.6	1.8	6.7	7.0	—	13.7	0.8	3.4	5.0	5.8

a) Highly suspect data: values unrealistically high.



### Elastomer Interactions With SHELLDYNE-H® Fuel

Three SHELLDYNE-H® fuel samples, which were exposed to different elastomers by Atlantic Research Corporation (contract F33615-69-C-1849), have now been tested further to determine any possible solution of elastomer or leaching of plasticizer during this exposure. In Figure 37 it is shown that the U.S. 941 Viton/Nomex nylon had the maximum effect on the light transmission properties of SHELLDYNE-H® fuel in the 335-375 millimicron wavelength range, although curiously the U.S. 3094 Viton gave the only significant change in refractive index as compared to the original stock (see Table 49). Results of microgum analyses, which indicate the amount of high boiling materials present, are shown in Table 49. These were run at 260°C under vacuum, at which conditions about 0.8 mg. carbon/100 g sample was obtained with the unexposed fuel. Fuel exposed to either U.S. 941 Viton/Nomex nylon or U.S. 3094 Viton gave about double this amount, but only the sample in contact with U.S. 566 Nitrile Nylon gave markedly higher results (20 times as much).

Table 49. MICROGUM AND REFRACTIVE INDEX ANALYSES OF SHELLDYNE-H® FUEL  
SAMPLES SOAKED WITH ELASTOMERS FOR A PERIOD OF SIX MONTHS

Elastomer	Microgum (expressed as carbon) mg/100g SHELLDYNE-H® Fuel	$n_D^{20}$
none	0.8, 0.9	1.5396
U.S. 941 Viton/Nomex Nylon	1.6, 1.8	1.5394
U.S. 3094 Viton	1.6, 1.9	1.5385
U.S. 566 Nitrile Nylon	15.6, 16.6	1.5394

Thus, three different methods have each selected a different elastomer as the one having the most interaction with SHELLDYNE-H® fuel. Nitrile nylon apparently contributes the most heavy material to SHELLDYNE-H® fuel, although this material apparently has a smaller effect upon light transmission and refractive index than do the impurities from the other two elastomers. This suggests the possibility of micelle or suspended solids formation in the Nitrile nylon sample, which visual inspection in room light confirmed. Both a yellow tint and a slight turbidity were observed. The other three samples were all water white and free of apparent solids content. Since neither micelles nor solids affect transmission of light of short wavelength appreciably, the more minor effect of the Nitrile nylon on refractive index and light absorption is at least partially explained.

In a final series of tests, the four samples of SHELLDYNE-H® fuel of Table 49 were run by GLC analysis. The three samples which had been soaked with elastomers all showed a small unidentified peak representing about a 0.1 percent impurity, which was not present in the original stock. Other than that, the elastomer soaked SHELLDYNE-H® fuel samples all gave similar GLC analyses. The impurity could either have been introduced to the original material in handling prior to soaking, or might have come from the polymer film (which appeared to be polyethylene or polypropylene) which had been placed over the shipping bottles prior to capping.

Figure 37 follows

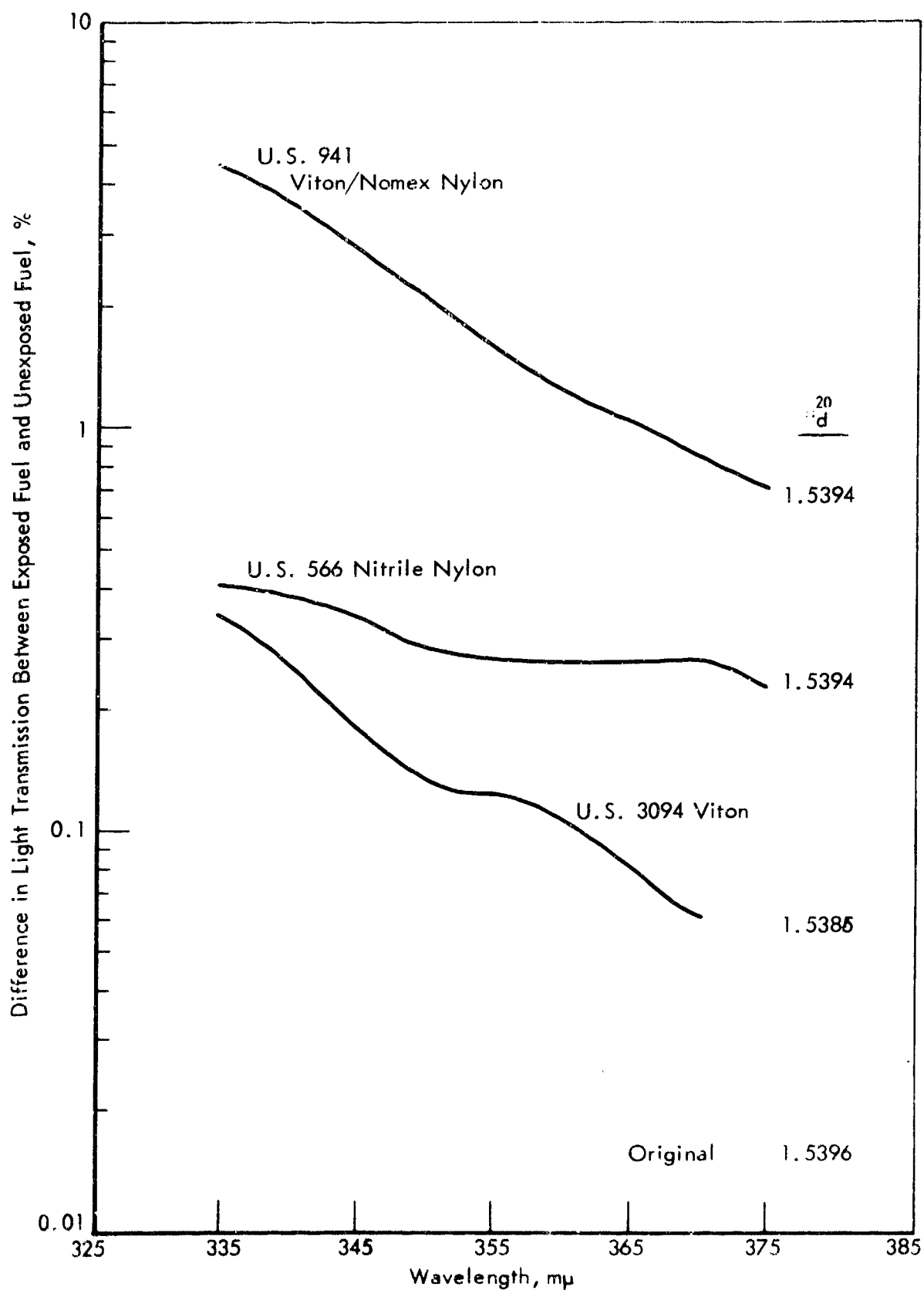


Figure 37. EFFECT OF EXPOSURE OF SHELLDYNE-H<sup>(®)</sup> FUEL (F-157)  
TO DIFFERENT ELASTOMERS

Whatever effect this film might have had, it probably would have been the same for all three elastomer soaked samples. The fresh SHELLDYNE-H<sup>®</sup> fuel came from our own retainer stock and was not exposed to the film. For the GLC procedure used, in which the sample size was 1 microliter, the detection limit is about 0.01 percent, unless the small impurity peaks are close to one of the larger peaks, in which case sensitivity would be much less.

In summary, we feel that the microgum test gives the more significant results with regard to compatibility of these elastomers with SHELLDYNE-H<sup>®</sup> fuel. From previous experience with SHELLDYNE-H<sup>®</sup> fuel it has been shown that light transmission properties do not necessarily correlate with coker test results. The reduction in light transmission indicates the presence of trace contamination, but thermal stability tests would be required to demonstrate any adverse effect on the fuel.

#### THERMAL STABILITY OF NEW BATCH OF METHYLCYCLOHEXANE

A new batch of 1200 gallons of MCH has been made by hydrogenation of toluene, and on checking this material for thermal stability it was found to be defective when compared to the MCH remaining from the last production run. It appeared that the cause of the poor thermal stability ratings was due to presence of suspended nickel catalyst particles, although this is surprising since the MCH had been distilled. Refractive index determinations of the twelve drums of product showed no variations whatever. SD/M-7 coker tests at 475°F demonstrated that silica gel treatment brought the fuel up to standard and that filtration through a 0.45-micron membrane filter gave even greater improvement. Thus, two methods for upgrading were available. We are now, therefore, filtering the MCH through a 0.2-micron membrane filter, and will blend the new material with the MCH now in the storage tank as soon as coker tests confirm a high thermal stability. This method of upgrading is comparatively simpler and cheaper than that of silica gel treatment.

#### ESTIMATION OF PHYSICAL PROPERTIES OF FUELS

##### JP-7 Jet Fuel

Revised physical properties for JP-7 jet fuel (F-71) have been obtained using improved predictive methods and are given in the Appendix. These techniques were used in estimating physical properties for decalin and JP-5 jet fuel.<sup>3)</sup> The PVT behavior of the gas was represented by the Redlich-Kwong-Ackerman reduced equation of state:<sup>19)</sup>

$$Z = Z_{RK} + Z_1 + \omega Z_2 \quad (13)$$

where  $Z$  = compressibility factor

$Z_{RK}$  = compressibility factor calculated by the Redlich-Kwong equation of state<sup>20)</sup>

$\omega$  = acentric factor

$Z_1, Z_2$  = generalized functions of reduced pressure and temperature

The ideal gas heat capacity was represented by the group contribution method of Rihani-Doraiswamy:<sup>21)</sup>

$$c_p^\circ = a + bT + cT^2 + dT^3 \quad (14)$$

where  $c_p^\circ$  = ideal gas heat capacity  
 $a, b, c, d$  = parameters determined by group contributions

Most of the other gas properties were obtained from these two properties using thermodynamic relationships.

Chemical analyses indicated that JP-7 is composed primarily of paraffins in the  $C_{11}$  to  $C_{16}$  range. Hence, the critical properties of the normal paraffins in this molecular weight range were used to obtain pseudocritical properties for the jet fuel. Pseudocritical temperature and pressure were defined as

$$T_c = \sum_i y_i T_{ci} \quad (15)$$

$$P_c = \sum_i y_i P_{ci} \quad (16)$$

where  $y_i$  = mole fraction of component  $i$   
 $T_{ci}, P_{ci}$  = individual component critical properties

In addition, the acentric factor was obtained from experimental vapor pressure data, and the Rihani-Doraiswamy coefficients for the fuel were taken as molar averages of the coefficients for the individual normal paraffin components.

Liquid properties were revised in the following manner. Previous estimates of density were corrected to agree with an available experimental density at 60°F and the critical density as predicted by the equation of state. Liquid enthalpy and heat capacity, being based on the revised gas properties, were also revised:

$$H_l = H_g - \Delta H_v \quad (17)$$

$$c_{pl} = c_{pg} - \frac{d(\Delta H_v)}{dT} \quad (18)$$

where  $H_l, H_g$  = liquid and gas enthalpy, respectively  
 $c_{pl}, c_{pg}$  = liquid and gas heat capacity, respectively  
 $\Delta H_v$  = enthalpy of vaporization

Also, revised values of liquid thermal conductivity were obtained by the Robbins-Kingree correlation.<sup>22)</sup> Previous estimates of vapor pressure, enthalpy of vaporization, and liquid viscosity were left unchanged.

The revised properties are considered to be better than previous values,<sup>2)</sup> because the most recently proven correlations have been used and experimental data have been applied where possible. They agree well with published or predicted values for similar petroleum fractions. The greatest improvement has been made with liquid heat capacity and thermal conductivity. Comparison with heat transfer experiments made previously in the FSSTR<sup>3)</sup> showed that the new heat capacity values are far superior to the old values.

#### Binary Mixtures of Methylcyclohexane and a Low Molecular Weight Hydrocarbon

Physical properties were estimated for binary mixtures of methylcyclohexane (MCH) and a low molecular weight hydrocarbon. These properties are tabulated in the Appendix for mixtures containing ethane, propane, and n-butane. Mixture properties were estimated from known properties of the individual constituents.<sup>3,23)</sup>

The following properties were calculated as molar averages of the pure component properties:

- Pseudocritical Temperature
- Pseudocritical Volume
- Pseudocritical Compressibility Factor
- Acentric Factor
- Heat of Formation
- Net Heat of Combustion
- Liquid Specific Gravity
- Vapor Pressure

The above pseudocritical properties were used to calculate the pseudocritical pressure by the equation of state

$$P_c = \frac{RT_c Z_c}{V_c} \quad (19)$$

where

- $P_c$  = pseudocritical pressure
- $T_c$  = pseudocritical temperature
- $Z_c$  = pseudocritical compressibility factor
- $V_c$  = pseudocritical volume
- $R$  = universal gas constant

The liquid viscosity was calculated by a molar average of the logarithmic function of viscosity:

$$\ln \mu = \sum_i x_i \ln \mu_i \quad (20)$$

where  $\mu$  = viscosity of mixture  
 $\mu_i$  = viscosity of pure component i  
 $x_i$  = mole fraction of component i

The mixture enthalpy was estimated as the molar average of the pure component enthalpies at those conditions where both components are gases or liquids in their pure state. Nonideal mixing effects are not appreciable in these systems and were neglected in the calculations. At those conditions where the mixture is a gas dissolved in MCH, the calculated enthalpy was reduced to account for the heat of absorption of the gas.

The melting points for the mixtures are based on experimental data for mixtures of MCH and propane. Melting and freezing points were measured for MCH-propane mixtures of three different compositions (Figure 38). An equation of the Clausius-Clapeyron type can be used to relate composition and melting point:

$$\frac{1}{T_f} - \frac{1}{T_{fo}} = \frac{R}{\Delta H_f} \ln x_{MCH} \quad (21)$$

where  $T_f$  = melting point of mixture  
 $T_{fo}$  = melting point of MCH ( $-126.6^\circ\text{C} = 146.6^\circ\text{K}$ )  
 $\Delta H_f$  = heat of fusion of MCH  
 $x_{MCH}$  = mole fraction of MCH

$$K_f = \frac{RT_{fo}^2}{\Delta H_f} = \text{melting point depression constant} \quad (22)$$

Equation (21) can be approximated by

$$T_f - T_{fo} = -\frac{RT_{fo}^2}{\Delta H_f} (1 - x_{MCH}) \quad (23)$$

Linear regression of the data was used to determine empirical coefficients for Equations (21) and (23) with the following results. For Equation (21)

$$\frac{1}{T_f} - \frac{1}{T_{fo}} = -\frac{K_f}{T_{fo}^2} \ln x_{MCH} \quad (24)$$

where  $K_f = 35.14^\circ\text{K}$

For Equation (22)

$$T_f - T_{fo} = -K'_f (1 - x_{MCH}) \quad (25)$$

where  $K'_f = 40.86^\circ\text{K}$

If the known heat of fusion ( $16.43 \text{ cal/gm}^{24}$ ) is used in Equation (22), the theoretical melting point depression constant is found to be  $26.47^\circ\text{K}$ , quite different from the above values. Melting points predicted with this theoretical constant differ significantly from the data (Figures 38 and 39). Both Equations (24) and

Figures 38 and 39 follow

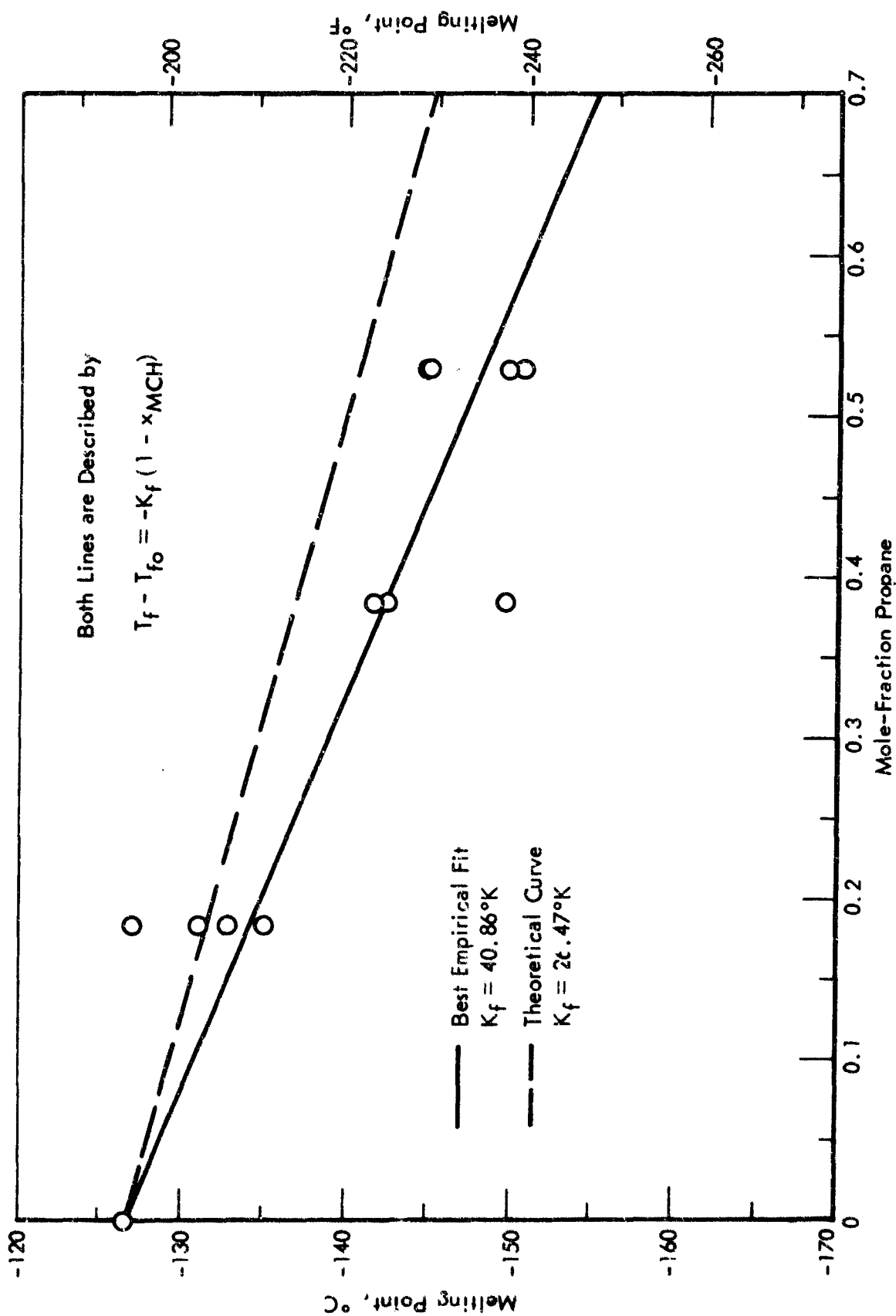


Figure 38 MELTING POINTS OF METHYLCYCLOHEXANE-PROPANE MIXTURES

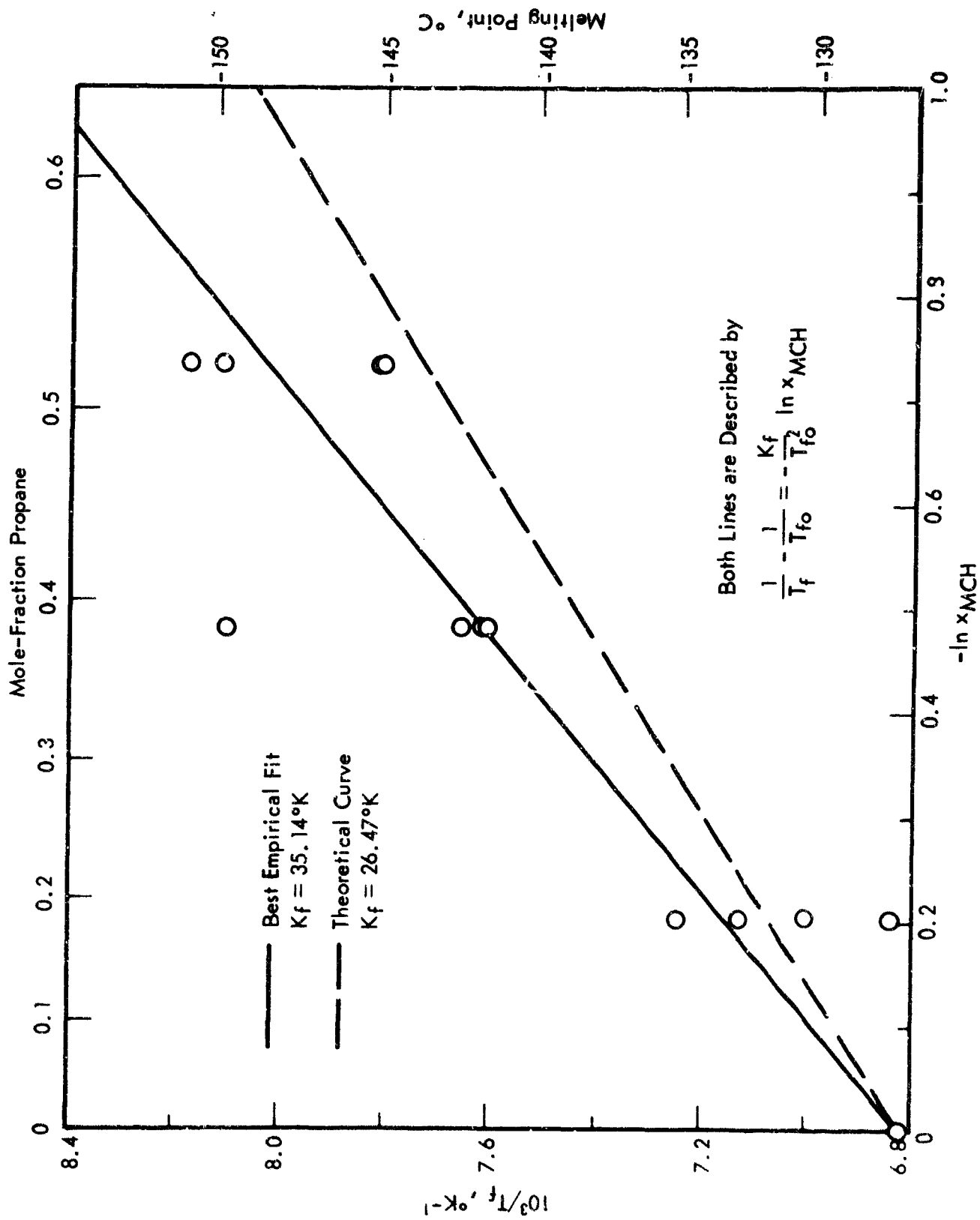


Figure 39. MELTING POINTS OF METHYLCYCLOHEXANE - PROPANE MIXTURES



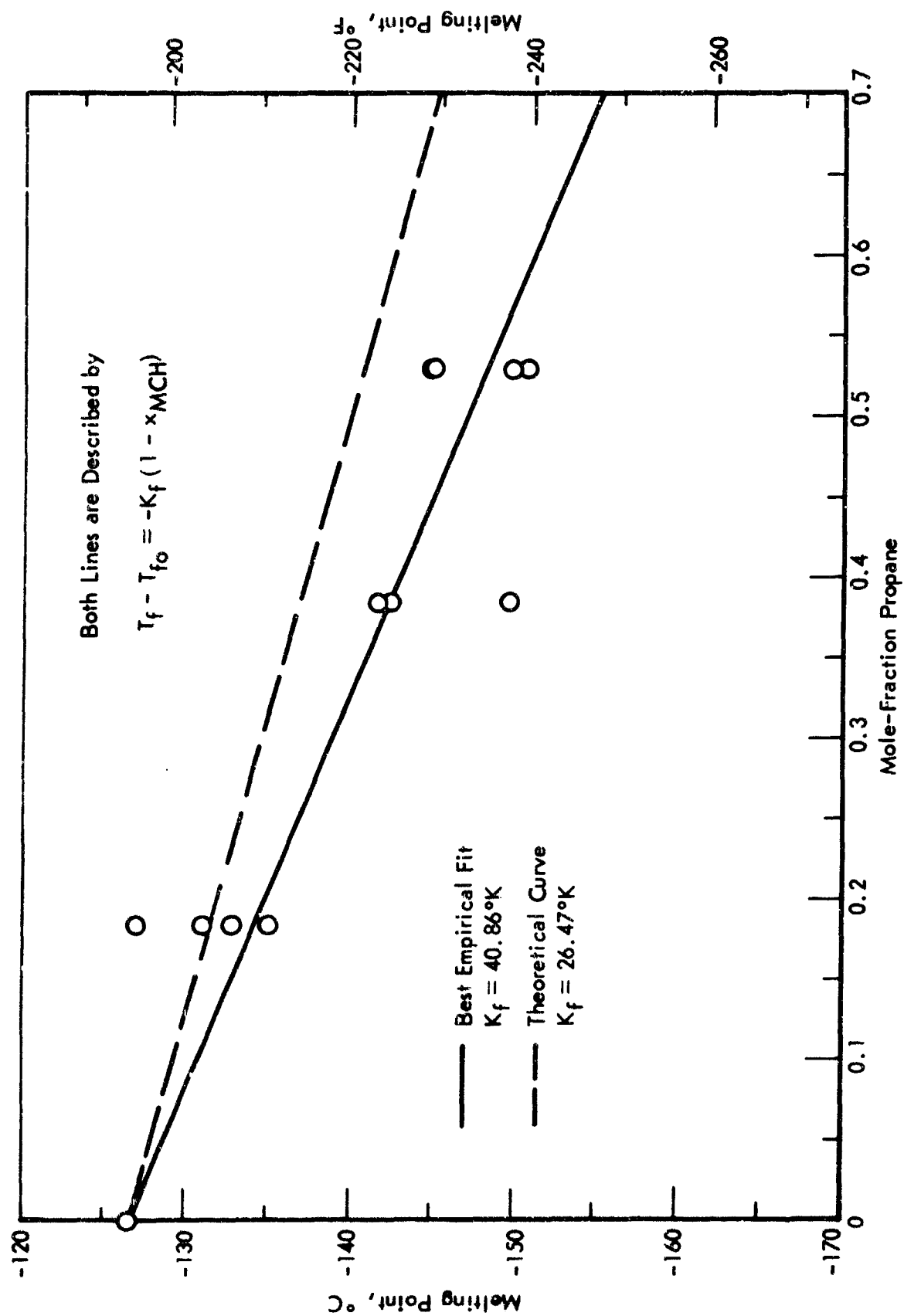


Figure 38. MELTING POINTS OF METHYLCYCLOHEXANE-PROPANE MIXTURES

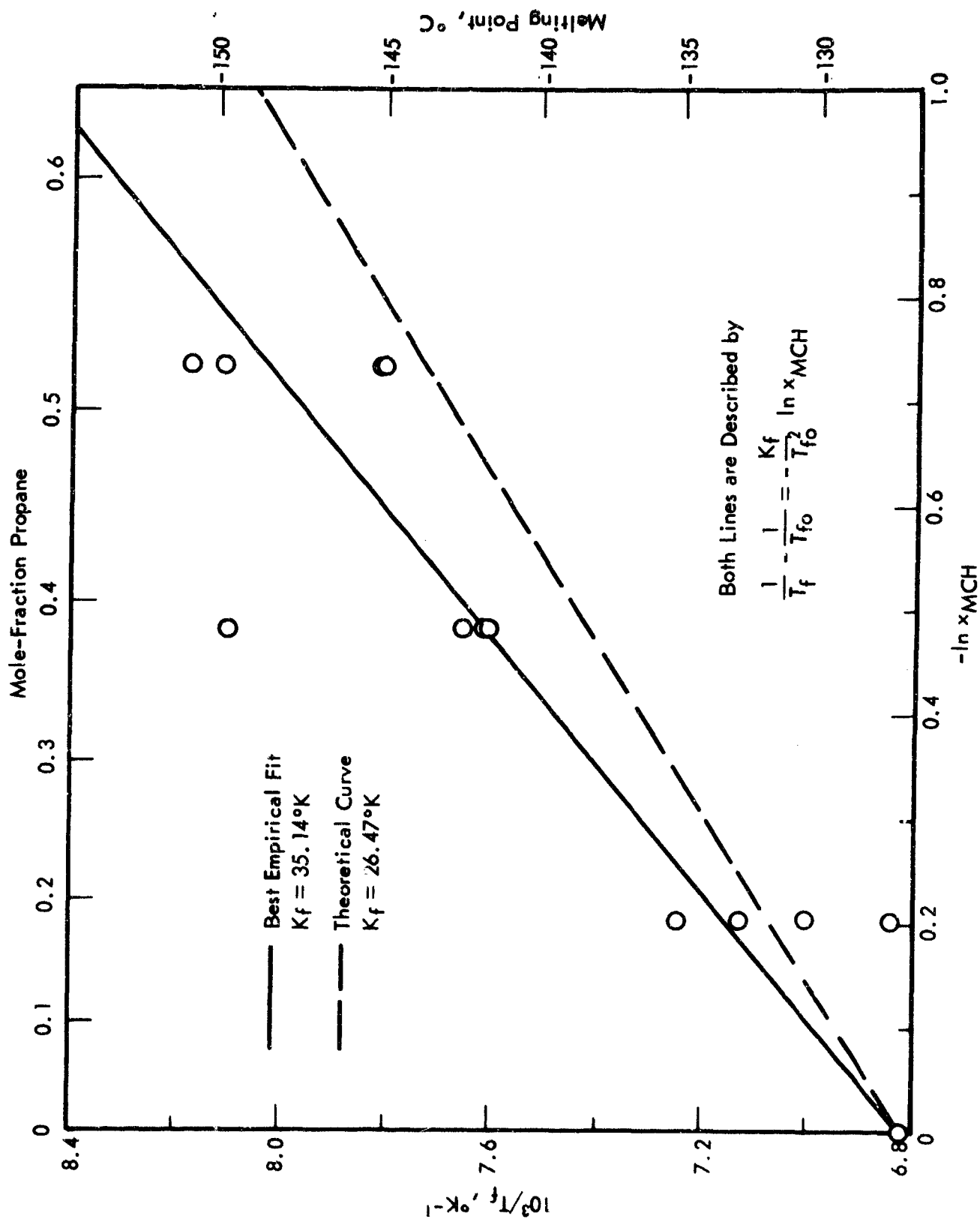


Figure 39. MELTING POINTS OF METHYLEYCLOHEXANE - PROPANE MIXTURES

(25) with the empirical coefficients lie within the accuracy of the data; they fit the data better than the theoretical predictions. Since Equation (25) is simpler, it was chosen to represent the melting points of low molecular weight hydrocarbons mixed with MCH. Calculated melting points are listed with the other physical properties of these mixtures in the Appendix.

#### SHELLDYNE-H® Hydrocarbon Fuel

The surface tension of SHELLDYNE-H® fuel has been measured at three temperatures by the du Nouy ring method. These data correlated quite well to fit

$$\gamma = a (T_c - T)^n \quad (26)$$

where  $\gamma$  = surface tension (dyne/cm)  
 $T$  = temperature (°C)  
 $T_c$  = critical temperature (530°C)  
 $a$  = 0.010  
 $n$  = 1.33

Measured and extrapolated values of the surface tension are given in Table 50 and Figure 40.

Table 50. SURFACE TENSION OF  
SHELLDYNE-H® FUEL

T, °C	Surface Tension (dyne/cm)	
	Measured	Predicted
0		42.0
25	39.4	39.4
50	36.7	36.8
75	34.3	34.3
100		31.8
200		22.4
300		13.8
400		6.5
500		0.9
530		0

#### SUPERSONIC COMBUSTION OF HYDROCARBONS

The methods used in obtaining and analyzing our supersonic combustion data have been re-evaluated. Some improvements in the equipment have been made. A new equation has been used to correlate the ignition delay times. Some new experimental data have been obtained. The rate of combustion of tetralin has been investigated.

Figure 40 follows

### Experimental Equipment and Conditions

Several problems have been encountered in operating the shock tube with heavier hydrocarbon fuels. The tube had to be heated to 40-80°C. The resulting thermal stresses were hard on the resistance gauges which were used to measure the velocity of the shock wave. As constructed, the gauges were somewhat fragile. They consisted of a thin layer of platinum which had been sputtered onto a quartz disc encased in Bakelite. The differences in thermal expansion and the structural weaknesses led to frequent cracking and shorting. A sturdier device was designed and built. A similar design was used but the Bakelite body was replaced with aluminum. The quartz disc was replaced with Grade A Lava ceramic glued into the body with an epoxy resin. The platinum film was painted on as Liquid Bright Platinum, Hanovia No. 05-X. The improved resistance gauges have proved to be more reliable and have about the same dynamic response as the more fragile gauges used previously.

The mechanism for puncturing the shock tube diaphragms had to be modified by replacing the gasket with one able to withstand higher temperatures. A nylon tip is now used on the plunger which has improved the life and reliability of the plunger.

In order to prepare gas mixtures containing naphthalene, the mixture preparation system was modified. The hydrocarbons investigated to date have been gaseous or liquid at ambient conditions. Naphthalene, being a solid, presented a problem. The solution was to mount a tube, which could be isolated and removed, above the mixture cylinder. A schematic diagram of the system is shown in Figure 41. With the cylinder heated and evacuated, and the tube cold and detached at valve No. 1, a weighed amount of naphthalene crystals is added to the tube. The tube is then fastened to valve No. 1 and is evacuated by opening valve No. 1 to vacuum. When the air has been withdrawn, valve No. 1 is closed, valve No. 2 is opened, and the tube is heated to vaporize the naphthalene and drive the vapors into the cylinder. The other gases are added to the cylinder through valves No. 1 and No. 2 in the normal way.

The experimental data obtained have been on those hydrocarbons involved in the dehydrogenation of decalin, namely decalin, tetralin, and naphthalene. A summary of the conditions of the experiments is presented in Table 51. The complete data on ignition delay times may be found in the Appendix. The data have been obtained primarily at 9 psia and 15 psia and temperatures from 1140° to 1760°K, resulting in ignition delay times from 40 to 4000  $\mu$ sec.

### Shock Tube Attenuation

An attempt was made to improve the shock tube results by accounting for velocity attenuation by measurement. The results have shown that assuming an attenuation coefficient of  $1 \times 10^{-3}$ , constant for all conditions, is the most reasonable procedure.

There are many non-idealities in shock tube flow. One of these is the decrease in the velocity of the shock wave as it moves down the tube, caused by the build-up of a boundary layer at the wall of the tube. A review of wall effects has been made by Enrich and Wheeler,<sup>25</sup> where they present the attenuation equation:

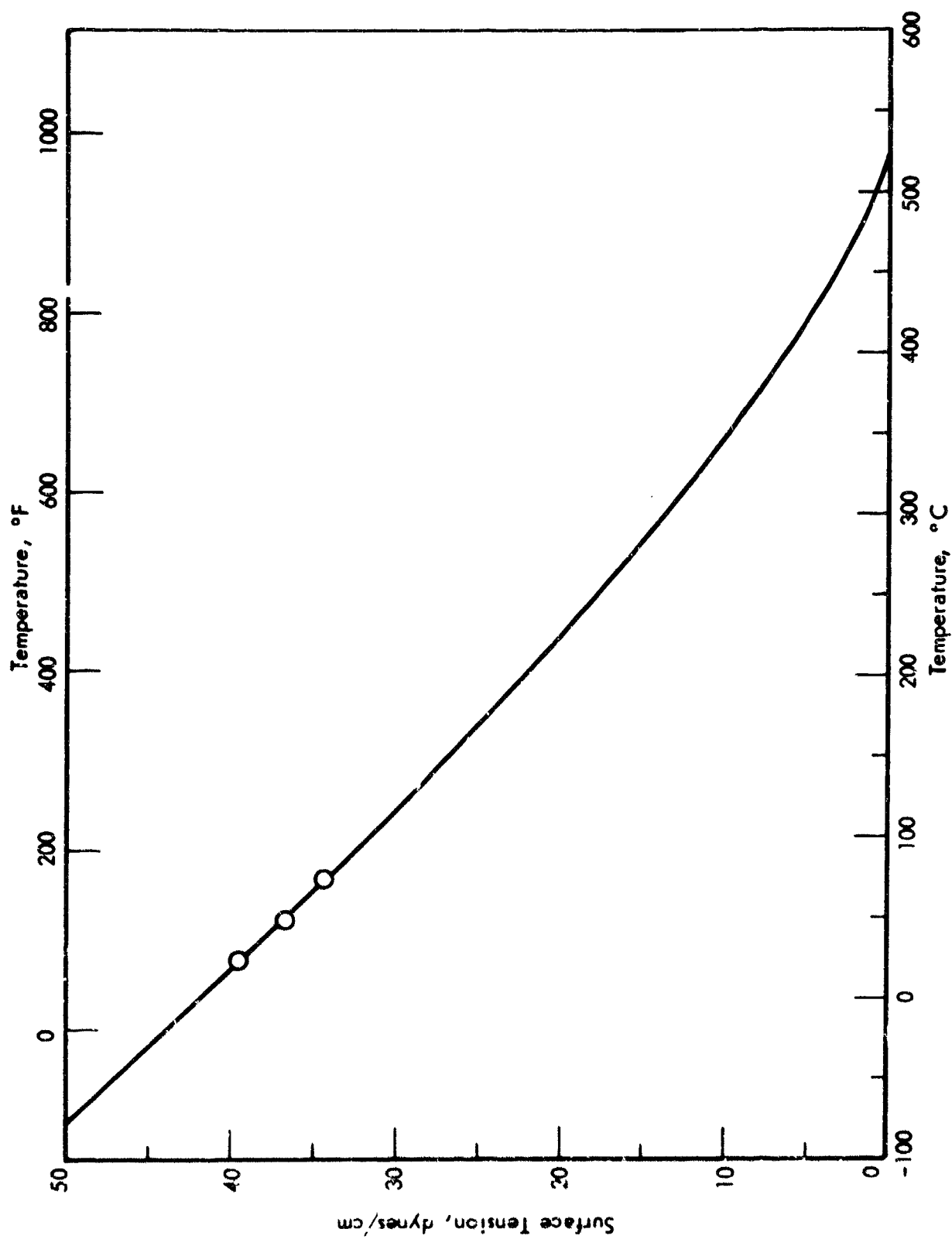


Figure 40. SURFACE TENSION OF SHELLDYNE-H<sup>®</sup> FUEL

Table 51. SUMMARY OF CONDITIONS OF SHOCK TUBE RUNS

Fuel	Equivalence Ratio*	Percent Argon
decalin	0.2	90
decalin	0.5	99
tetralin	0.1	99
tetralin	0.1	80
tetralin	0.11	90
tetralin	0.5	99
tetralin	0.57	90
tetralin	1.0	95
tetralin	1.0	99
tetralin	1.21	90
naphthalene	0.1	90

\*Equivalence ratio is defined as the actual fuel to oxygen ratio divided by the stoichiometric fuel to oxygen ratio.

$$(P_{21} - 1) = (P_{21} - 1)_{\text{ideal}} \exp(-A \frac{x}{r}) \quad (27)$$

where  $P_{21}$  = ratio of pressures before and after shock wave  
 $x$  = distance from diaphragm  
 $r$  = hydraulic radius  
 $A$  = attenuation coefficient

This equation is said to be accurate enough for most work. We have presented the same equation in a slightly different form:<sup>3)</sup>

$$(M_s^2 - 1)_2 = (M_s^2 - 1)_1 \exp(-A \frac{x}{r}) \quad (28)$$

where  $M_s$  = Mach number  
 $x$  = distance from point 1 to point 2.

Several experiments were performed to determine experimental values of attenuation coefficients. To date, values of  $0.5 \times 10^{-3}$  and  $1 \times 10^{-3}$  have been used for  $A$ . These values were obtained from measurements in the literature on similar shock tubes. The attenuation coefficients determined experimentally on our shock tube were correlated with initial channel pressure and the ratio of driver pressure to channel pressure. The resulting correlation was:<sup>a)</sup>

$$A = \frac{1}{4}(0.00436 - 0.0000457 P_1 + 0.0000698 \frac{P_4}{P_1}). \quad (29)$$

where  $P_4$  = initial pressure of driver gas  
 $P_1$  = initial pressure of reaction mixture

A set of experiments has been performed measuring ignition delay times and velocity attenuation simultaneously. We are now in a position to compare three different ways of estimating attenuation and its effect on ignition delay time measurements: (1) using a constant value of the attenuation coefficient as in the past ( $A = 1 \times 10^{-3}$ ), (2) using the correlation for attenuation coefficient, and (3) using the measured velocity attenuation. The ignition delay times as determined by the three methods for the combustion of a decalin-oxygen-argon mixture are shown in Figures 42 to 44. Figure 44 shows that using the correlation is clearly unsatisfactory. A comparison of Figures 42 and 43 reveal that the other two methods are approximately equivalent. The general scatter in Figure 43 is slightly less, but three points are badly misplaced. Overall, using the constant value of attenuation coefficient gives the best results. The reason for this is not immediately clear. One would think that measuring the variations in attenuation and allowing the coefficient to change should improve the results and decrease the data scatter, not increase it.

a) We previously used the hydraulic diameter rather than radius. This introduces a factor of 4 into the correlation, i.e.,  $A' = 4A$ .

Figures 41 through 44 follow

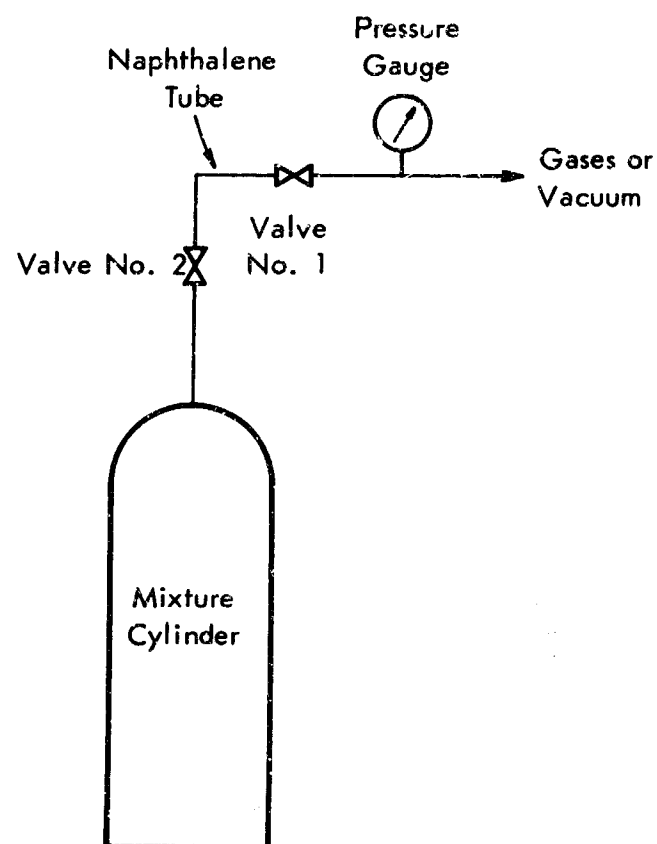


Figure 41. SYSTEM FOR PREPARATION OF GASEOUS COMBUSTION MIXTURES CONTAINING NAPHTHALENE



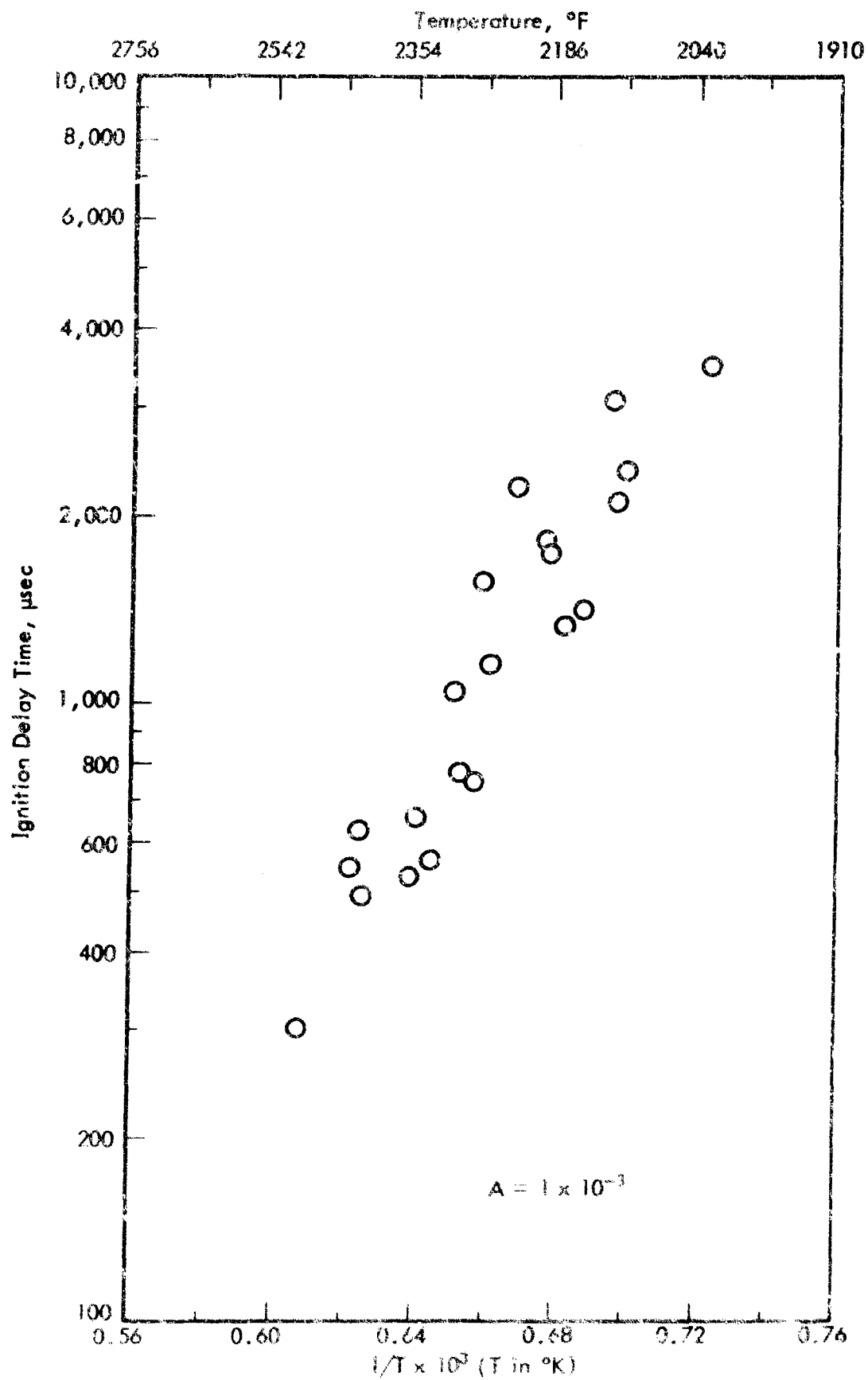


Figure 42. COMBUSTION OF DECALIN USING  
CONSTANT ATTENUATION

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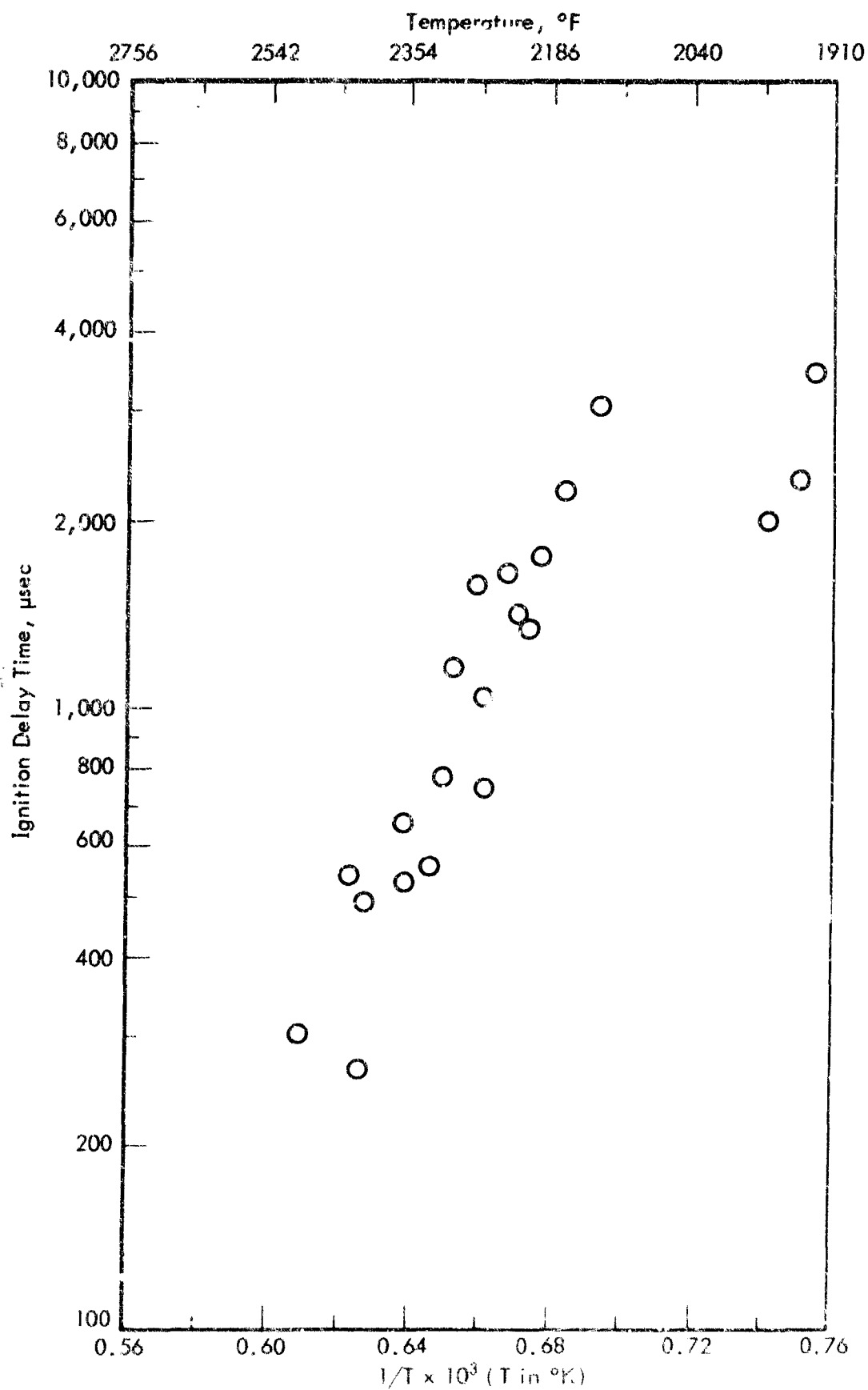


Figure 43. COMBUSTION OF DECALIN USING MEASURED ATTENUATION

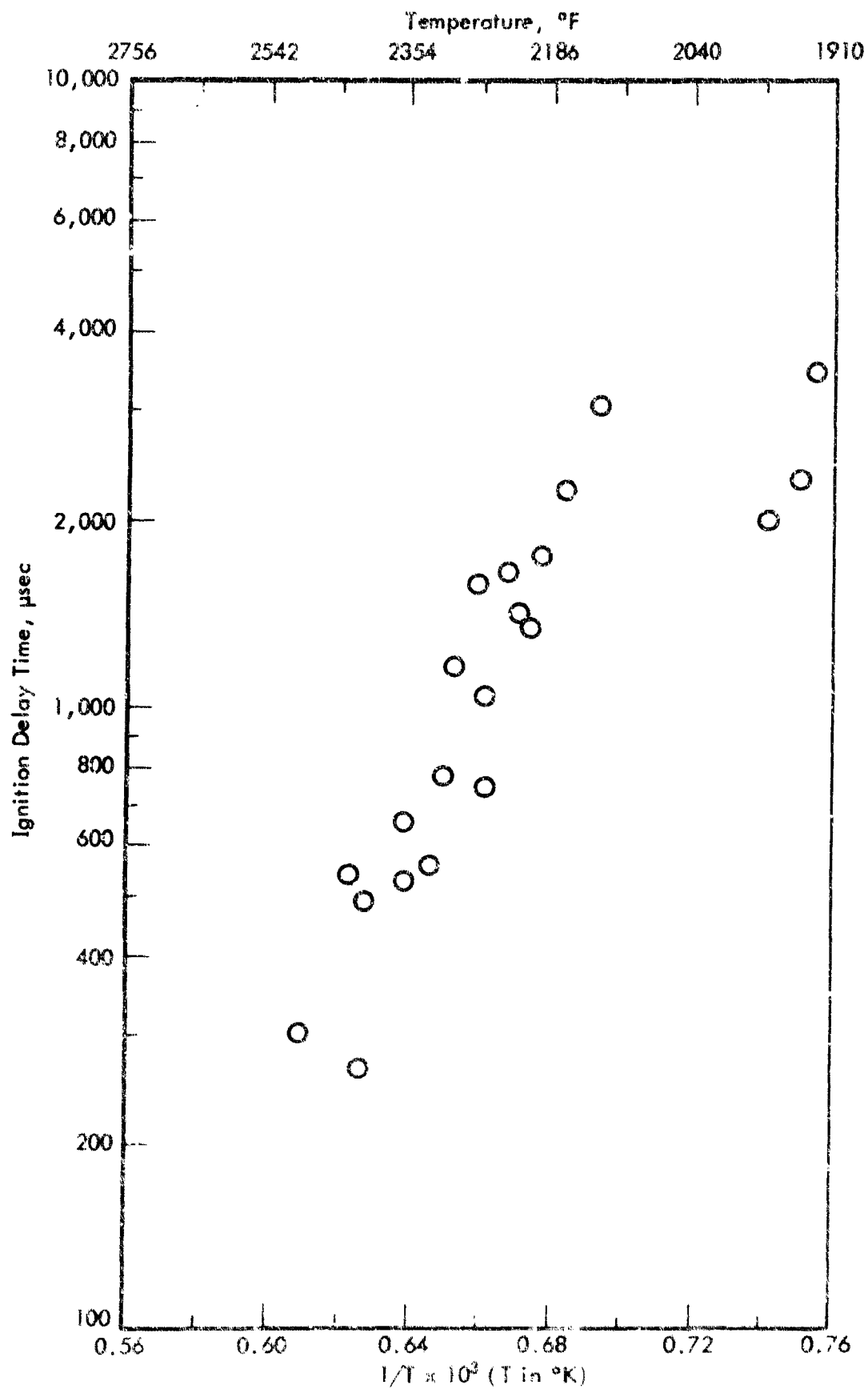


Figure 43. COMBUSTION OF DECALIN USING MEASURED ATTENUATION

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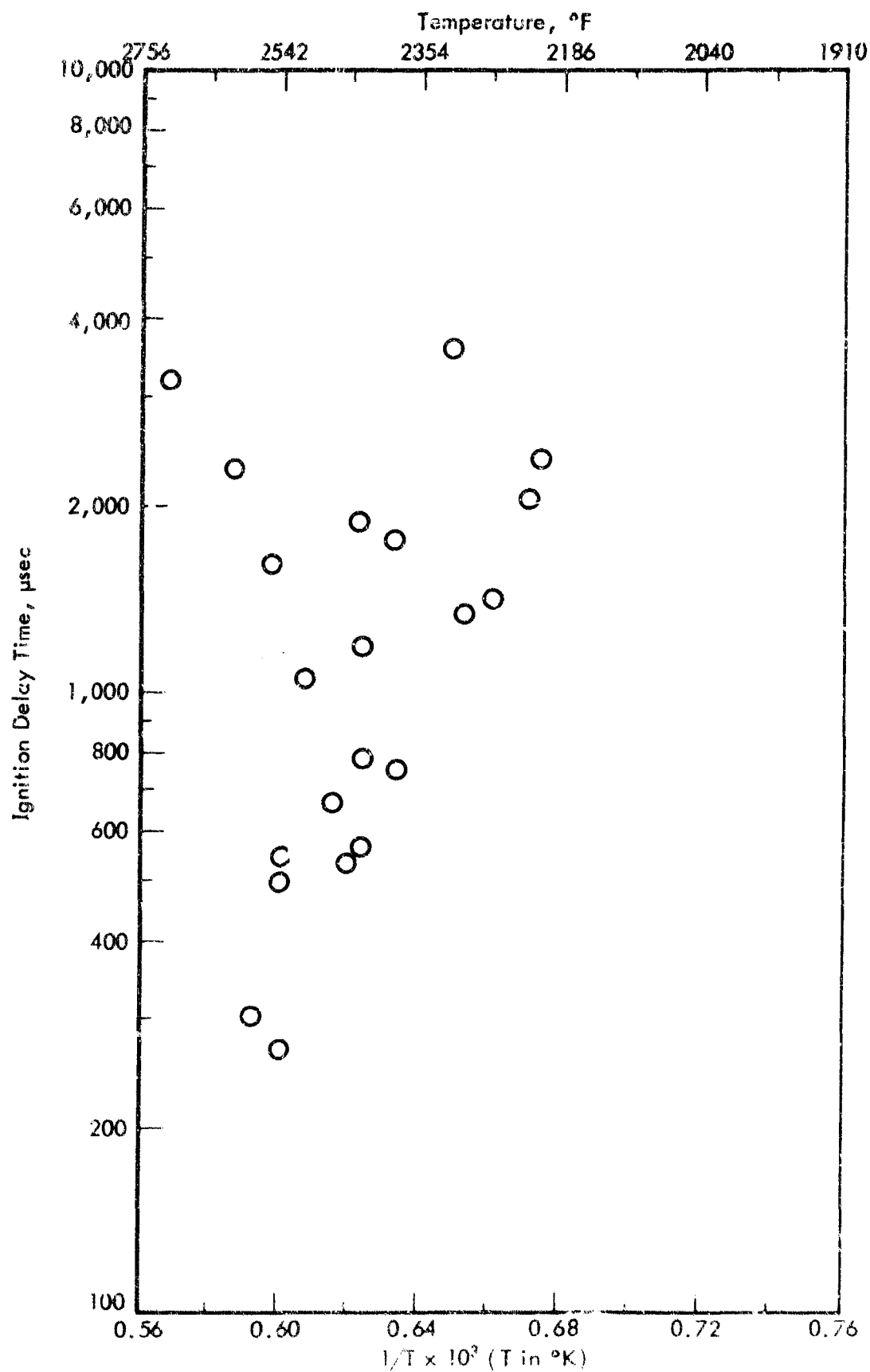


Figure 44. COMBUSTION OF DECALIN USING ATTENUATION CORRELATION

The explanation for this behavior appears to lie with the measurement of the shock wave velocity. Figure 45 shows a plot of average shock wave velocities at two points in the tube, in terms of the times of wave passage between point A and point B (18 inches) and between point B and point C (24 inches). The results do not fall precisely on a smooth curve, indicating either that the shock wave velocity fluctuates as it moves down the tube, or that there is appreciable error in the time measurements. A fluctuating shock wave velocity is fairly unlikely, although some variations can be caused by imperfect diaphragm breakage. The resolution of the time measurements is 1  $\mu$ sec, but the errors in measurement could be higher. Typical scatter of the data is 2-3  $\mu$ sec. The large effect of this apparently small scatter is shown by a plot of the attenuation coefficients calculated from the same data (see Figure 46). The values of A scatter between  $-0.09 \times 10^{-3}$  and  $1.7 \times 10^{-3}$ , and do not correlate with anything, including shock velocity. This is the reason for the failure of the correlation for attenuation. It is interesting that the mean value of all of the coefficients is  $0.99 \times 10^{-3}$ , which is remarkably close to the value of  $1 \times 10^{-3}$  that was estimated from literature data. If the measurement of shock velocity was accurate, one would expect that using the measured values of A would reduce the scatter of ignition delay times. Since this improvement was not found, the problem must lie in the measurement of shock velocity. Efforts to improve velocity measurements would involve the purchase of more accurate electronic instrumentation and more instruments to measure the velocity at several points in the shock tube. This is probably not worth the expenditure of time and money required. Therefore, the best that can be done is to take the average value of A, determined experimentally to equal  $1 \times 10^{-3}$ , and use it as a constant in all calculations.

#### Correlation of Ignition Delay Times

A new correlating equation has been used to represent the experimental data on ignition delay times:

$$\ln \tau = b_0 + b_1 \ln (c_{O_2}) + \frac{E}{RT} + b_2 \ln (c_{Fuel}) \quad (30)$$

where  $\tau$  = ignition delay time,  $\mu$ sec  
 $b_0, b_1, b_2, E$  = correlating parameters  
 $c_{O_2}$  = oxygen concentration, gmole/liter  
 $R$  = gas constant,  $1.987 \times 10^{-3}$  kcal/gmole- $^{\circ}$ K  
 $T$  = absolute temperature,  $^{\circ}$ K  
 $c_{Fuel}$  = fuel concentration, gmole/liter

The methods of estimating the parameters have been investigated, as have the statistical techniques which are used to interpret the results.

The equation used to correlate the data up to now has been the same as Equation (30) without the last term, which describes the effect of the fuel composition. There was some question about the correlation for decalin, since it appeared that fuel concentration had some effect on the ignition delay times. So the decalin data were fit using Equation (30) plus an additional term describing

the effect of pressure. The standard errors of the equation obtained by fitting with the addition of one term at a time are shown in Table 52. As can be seen, the improvement by including the effect of fuel concentration is substantial, while only slight improvement is found by including the pressure effect. Therefore, it was decided to drop the pressure effect from the correlation but retain the fuel effect, resulting in Equation (30) as the final correlating equation.

Table 52. ERRORS OF DIFFERENT EQUATIONS OF DECALIN  
IGNITION DELAY TIMES

Equation	Standard Error of ln $\tau$
$\ln \tau = b_0$	0.87
$\ln \tau = b_0 + \frac{E}{RT}$	0.81
$\ln \tau = b_0 + \frac{E}{RT} + b_1 \ln(c_{O_2})$	0.68
$\ln \tau = b_0 + \frac{E}{RT} + b_1 \ln(c_{O_2}) + b_2 \ln(c_{Fuel})$	0.50
$\ln \tau = b_0 + \frac{E}{RT} + b_1 \ln(c_{O_2}) + b_2 \ln(c_{Fuel}) + b_3 \ln(P)$	0.48

In addition to the equation to be used, there is some question as to the best way to fit the data and the best way to weight the data. Equation (30) could also be written as

$$\tau = \exp(b_0)(c_{O_2})^{b_1}(c_{Fuel})^{b_2}\exp\left(\frac{E}{RT}\right) \quad (31)$$

the obvious difference being that in this form the parameters cannot be determined by linear regression. However, there is another difference. The fitting techniques used here seek the least sum of squared deviations between the observed values and the calculated values. Using Equation (31) and equal weighting to fit the data would minimize the sum of  $(\tau_{obs} - \tau_{calc})^2$  for all the data. Using Equation (30) (logarithmic form) would minimize the sum of  $[\ln(\tau_{obs}) - \ln(\tau_{calc})]^2$  for all the data. The resulting equations will be different. The logarithmic form will tend to emphasize short ignition delay times, whereas Equation (31) treats all of the data as being equally important and equally accurate. It is not obvious which method is the proper one. The ignition delay data for decalin were fit using the logarithmic equation, Equation (30), using standard linear regression techniques. The results are shown in Figure 47 as the observed ignition

Figures 45 through 47 follow

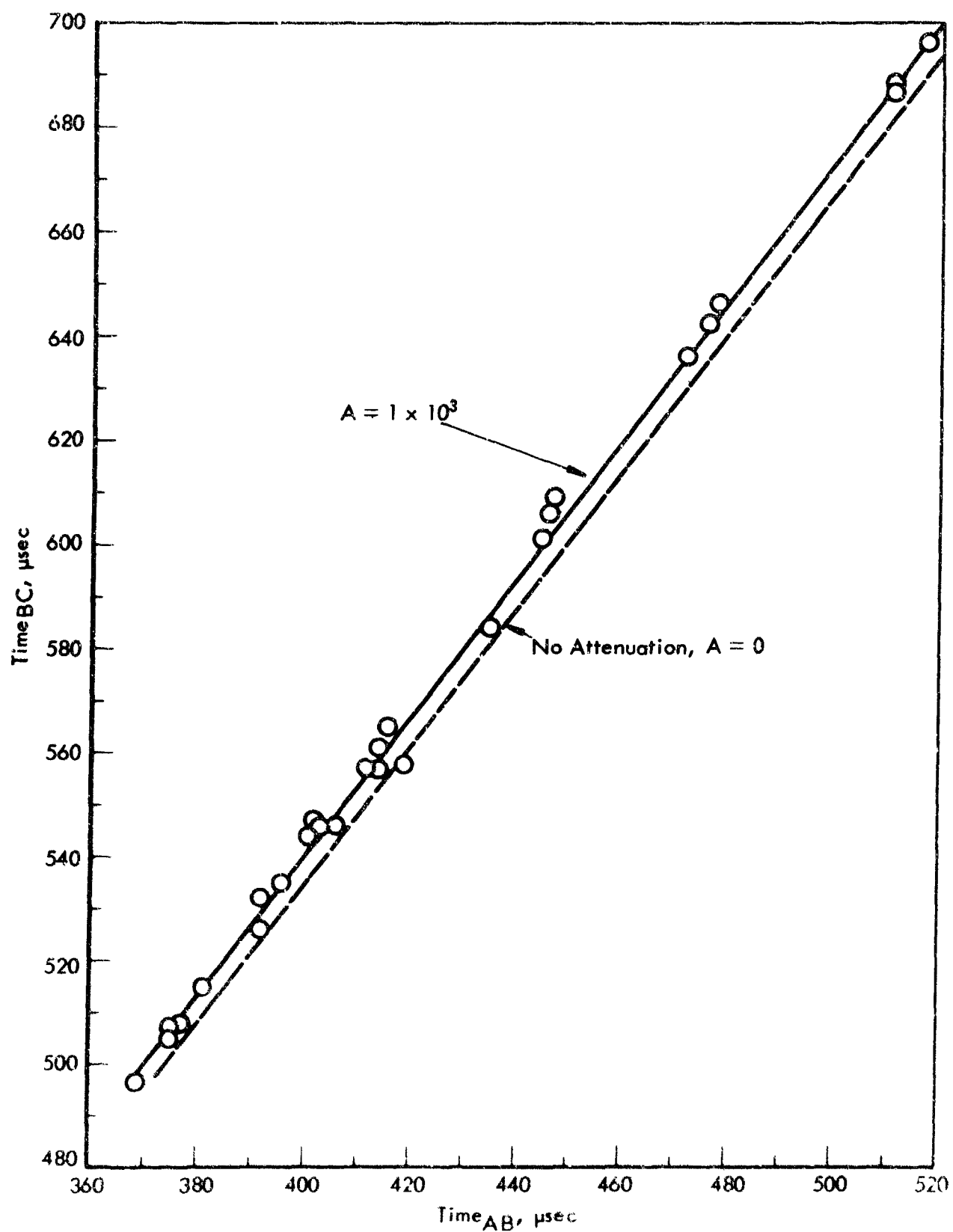


Figure 45. CORRELATION OF SHOCK WAVE VELOCITIES AT TWO POINTS IN TUBE

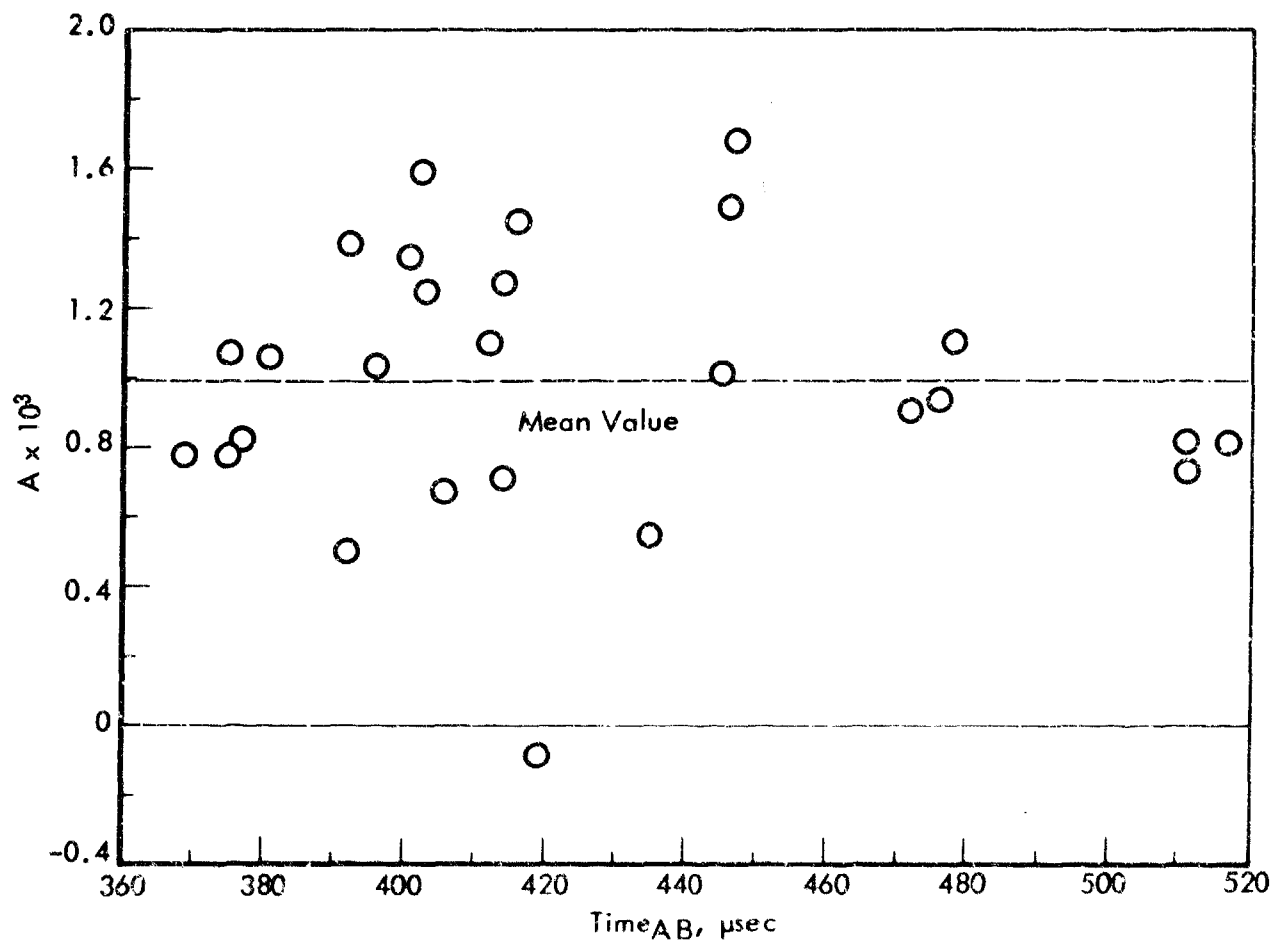


Figure 45. MEASURED ATTENUATION COEFFICIENTS



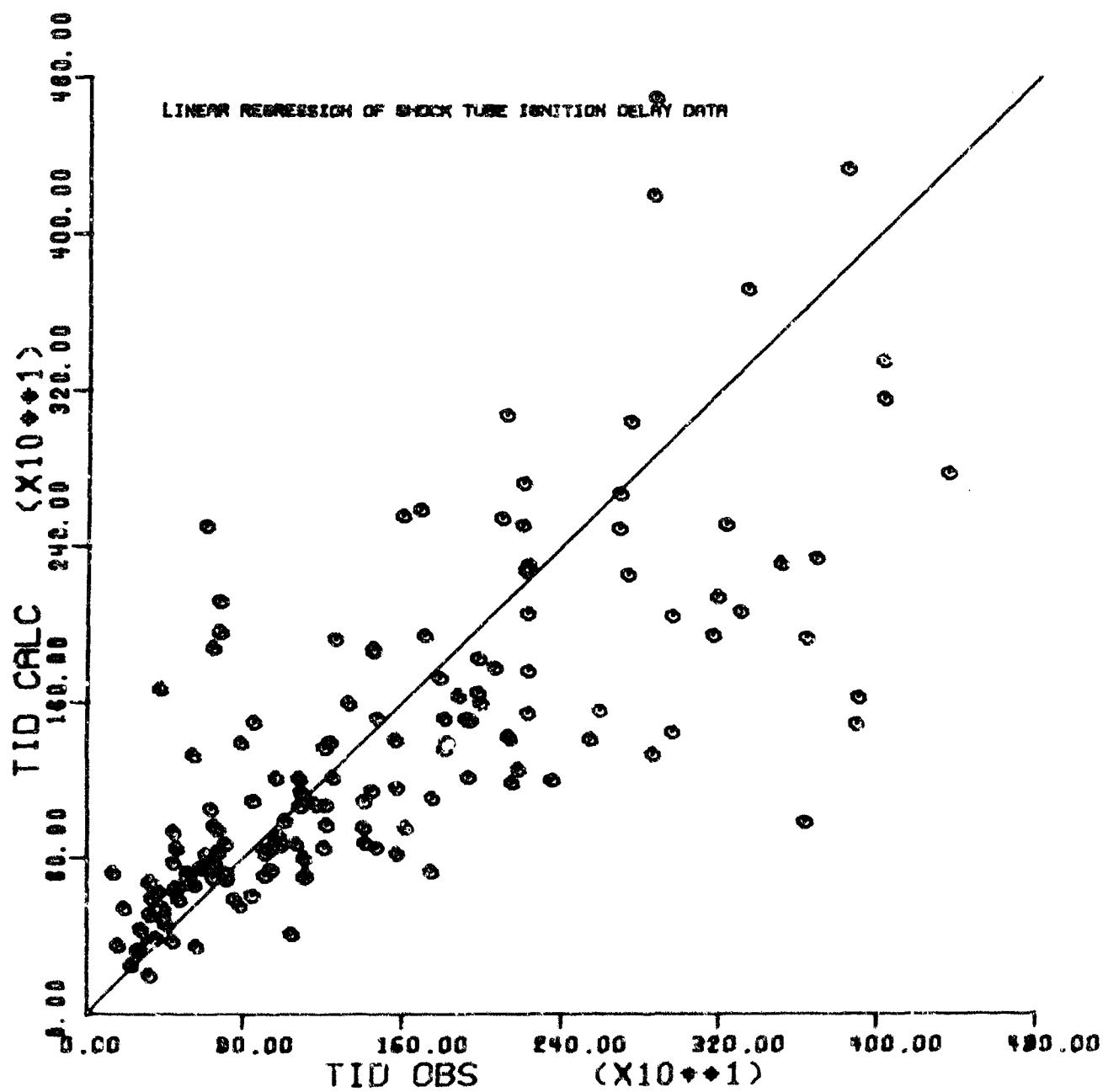


Figure 47. DECALIN DATA FITTED TO LOGARITHMIC EQUATION

delay times plotted against the calculated ignition delay times. The regression programs were set up to make these kinds of plots automatically. The correlation using equation (31) determined by nonlinear regression<sup>a)</sup> is shown in Figure 48. The graphs show the expected result; the linear regression gives a better fit at short times and a poorer fit at long times. Also apparent from both plots is that the errors increase as the delay times increase. The problem was rerun using Equation (31) assuming that the variance of the data increased as the times increased. The results are shown in Figure 49. The plot shows that the short times are fit better and the long times much poorer than the other two methods. Since we are primarily interested in short delay times for supersonic engine applications, one of the good fits at short times should be used. On the other hand, the correlation should not almost totally disregard the long delay time data as the increasing variance method does. Therefore, the logarithmic form, Equation (30), using linear regression was chosen as a compromise method for correlating the data. The values of the parameters found by the three methods are given in Table 53.

Table 53. METHODS OF IGNITION DELAY DATA CORRELATION

Regression Method	b <sub>0</sub>	b <sub>1</sub>	b <sub>2</sub>	$\frac{E}{(\frac{\text{kcal}}{\text{gmole}})}$
Linear, Logarithmic Form	-16.9	-1.27	0.79	55.7
Nonlinear, Constant Variance	-10.7	-1.05	0.74	44.2
Nonlinear, Changing Variance	-20.6	-1.18	0.49	55.0

Correlating equations and experimental data can be analyzed statistically with certain restrictions on the behavior of the errors. The most common approach is to assume that the errors are normally distributed and have a constant variance. Based on these assumptions, standardized techniques are available to determine the significance of the parameters, the significance of the correlation, the confidence limits of the parameters, and the confidence limits of the calculated values. As has already been mentioned, however, the errors in the correlation of the decalin data did not have constant variance. The other assumption was checked by performing a simple test to see if the errors were normally distributed. A probability plot was made of the residuals (errors) of the correlation of the data for tetralin. The residuals were plotted as  $\left( \frac{r_i - \bar{r}}{n} \right)$ , where  $r_i$  is the residual of the  $i$ -th

data point,  $\bar{r}$  is the mean residual, and  $n$  is the total number of points. The

a) A proprietary computer program using a modified form of the damped least squares method of nonlinear estimation was used.

Figures 48 and 49 follow

values of this quantity were arranged in decreasing order and plotted at equal percentage intervals on a normal distribution scale. If the result is a straight line, then the errors are normally distributed. The result for the tetralin data using Equation (30) is shown in Figure 50. Clearly, the errors are not distributed normally. The results are similar for other fuels. This means that the standard statistical tests do not strictly apply. To see if the form of Equation (30), which involves a logarithmic transformation, caused the un-normality, the data were refit by nonlinear regression to Equation (31). The probability plot of the residuals is shown in Figure 51. The errors still are not normally distributed, so that the problem is not with the fitting method or form of the equation. Since the normality assumption does not hold and the variance is not constant, the standard statistical tests cannot be used. The shape of the probability plots also do not resemble any other common distribution, such as logarithmic, exponential, or chi-square type. As a result, no estimate of confidence limits or significance will be meaningful. Strictly as an approximation one can calculate the standard deviation (standard error) of the calculated values assuming that the errors are normally distributed. This quantity is defined as the square root of the ratio found by dividing the sum of squared residuals by the number of degrees of freedom (number of data minus the number of parameters).

The correlating equation was fit to the ignition delay data for many of the systems studied previously. Table 54 summarizes the results. The parameters for the improved correlation are presented, along with the range of delay times studied experimentally and the standard error of each correlation. The new data on decalin and tetralin have been included in the correlations. The new decalin data were not available when the various fitting procedures were investigated; therefore, the parameters for decalin presented in Table 54 are not the same as those in Table 53. Sufficient data have not been obtained with naphthalene to enable correlation. The numerical values of the parameters probably have little significance. The parameters  $b_0$  and  $E$  are highly correlated with each other so that larger values of  $b_0$  are associated with smaller values of  $E$  and vice versa. It does appear that since all of the values of  $b_1$  are negative, oxygen has a beneficial effect by reducing ignition delay time, while for the most part, fuel seems to have a detrimental effect. Oxygen concentration seems to have a larger effect than fuel concentration, too. The values of  $E$  tend to fall into two ranges. Most of the fuels have values of about 40-50 kcal/gmole, while methylcyclohexane and its dehydrogenation products have values of about 20-30 kcal/gmole. The significance of this, if any, is not known. Figure 52 shows a plot of the correlations for a typical oxygen concentration and a stoichiometric fuel concentration. The uncertainty is typified by the standard error for the decalin correlation, which is shown graphically as the arrows in the margin of the figure. Figures 53 through 62 show plots of the fits to the data. The observed ignition delay times are shown against the calculated times.

#### Rate of Combustion of Tetralin

The data on the combustion of tetralin have been analyzed to obtain rates of combustion. Some trends seem evident but more experiments are indicated. Tetralin is an intermediate in the dehydrogenation of decalin to naphthalene. It would generally be found in the presence of hydrogen, but its combustion behavior is of interest both with and without hydrogen. The data on the combustion of tetra-

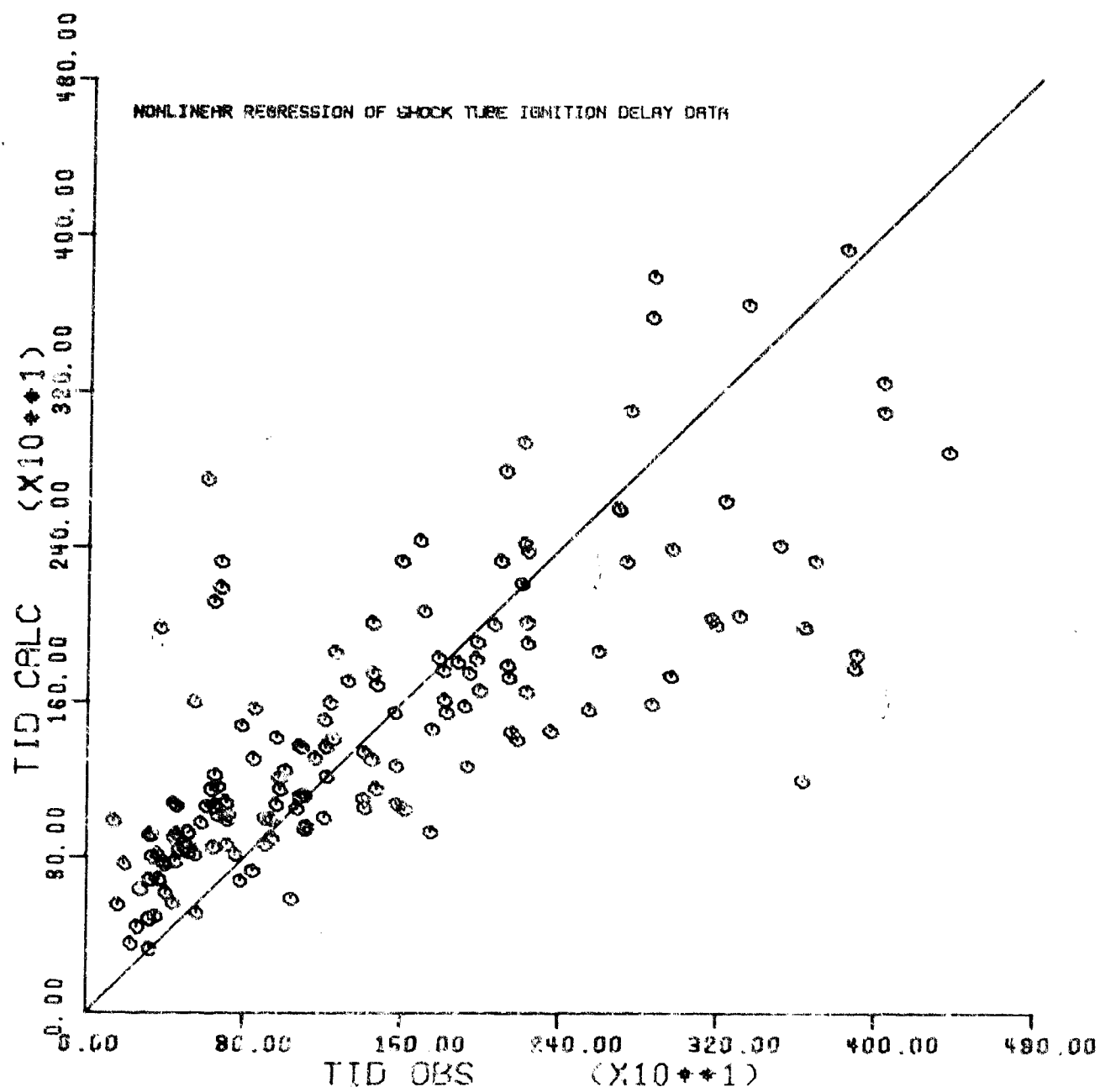


Figure 48. DECALIN DATA FITTED TO EXPONENTIAL EQUATION  
WITH EQUAL WEIGHTING (CONSTANT VARIANCE)

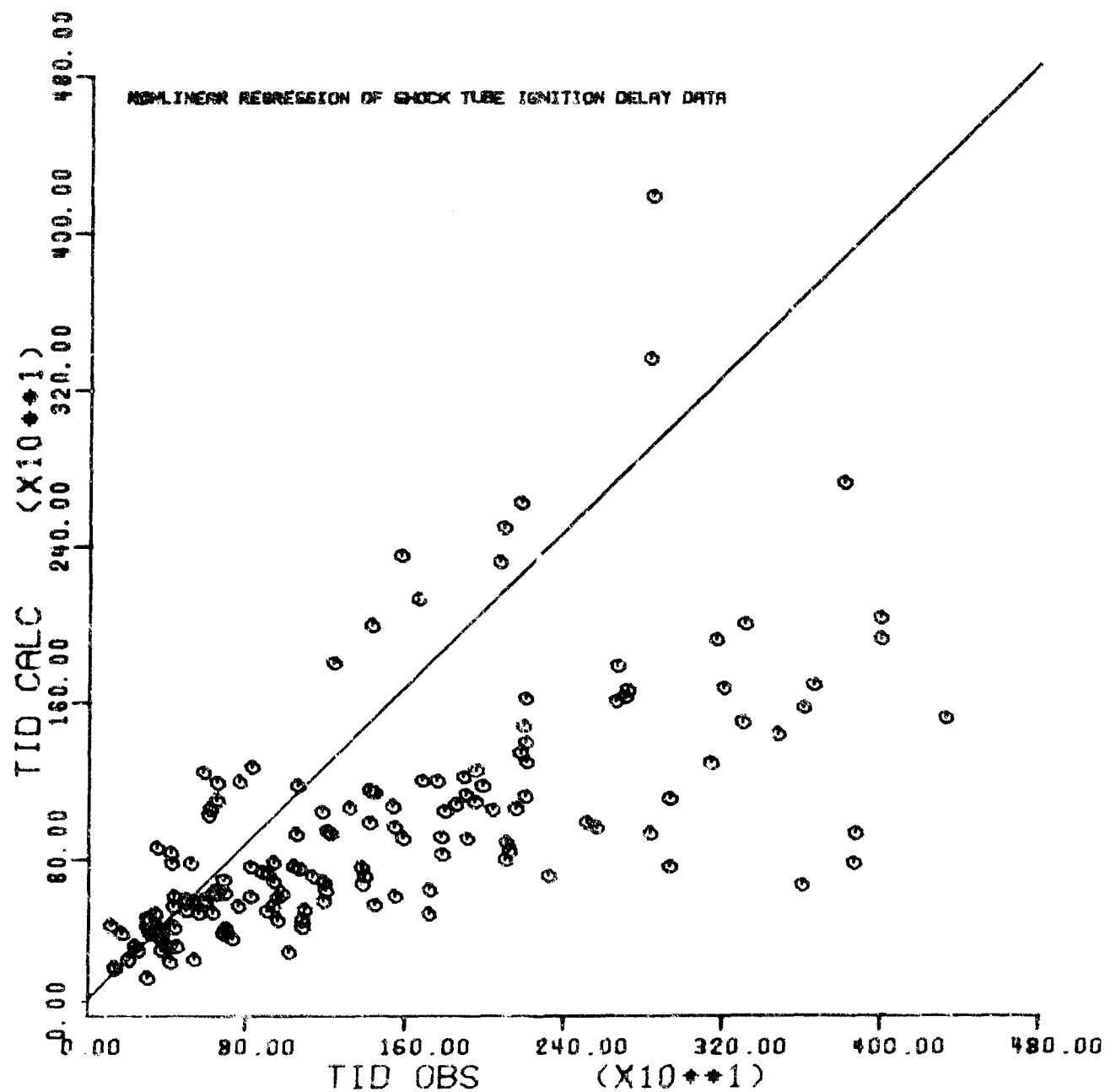


Figure 49. DECALIN DATA FITTED TO EXPONENTIAL EQUATION  
WITH CHANGING VARIANCE

Table 54. CORRELATIONS OF IGNITION DELAY TIMES

Fuel	b <sub>0</sub>	b <sub>1</sub>	b <sub>2</sub>	E	Range of $\tau$ ( $\mu$ sec)	Standard Error of $\ln \tau$
n-octane	-14.75	-0.76	0.15	41.1	70-4700	0.64
methylcyclohexane	- 2.89	-0.61	0.29	20.1	50-4100	0.61
toluene	- 5.84	-0.69	0.32	28.7	100-3400	0.39
toluene + H <sub>2</sub>	- 7.03	-0.68	0.16	26.2	100-4000	0.38
MCH + toluene + H <sub>2</sub>	- 5.36	-0.64	0.14	21.4	70-4400	0.58
dimethanodecalin	-19.56	-0.68	-0.33	45.8	80-4100	0.62
SHELLDYNE-H <sup>®</sup>	-13.80	-0.44	-0.28	35.6	60-3600	0.66
SHELLDYNE <sup>®</sup>	- 8.64	-0.94	0.60	41.0	80-3500	0.43
decalin	-10.94	-1.27	0.93	48.1	60-4300	0.50
tetralin	-15.99	-1.31	0.41	47.2	40-3800	0.68

Figures 50 through 62 follow

lin for ignition delay time measurements were analyzed to obtain combustion rates. The rate of combustion can be approximated by

$$r = \frac{dc}{dt} = k (c^* - c) \quad (32)$$

where  $r$  = the rate of appearance of  $CO_2$   
 $c^*$  = the ultimate concentration of  $CO_2$   
 $c$  = the current concentration of  $CO_2$   
 $k$  = the first order rate constant for combustion

Equation (32) can be integrated to give the reduced concentration of  $CO_2$  as a function of time:

$$\ln \frac{c}{c^*} = -kt \quad (33)$$

It is convenient to estimate  $k$  by finding the time required for  $c$  to reach half of the final value,  $c^*/2$ . Then  $k$  is found simply by

$$k = \ln 2/t_{1/2} \quad (34)$$

Since the mixtures which were burned at lower temperatures encountered the reflected rarefaction shock wave before combustion was complete, much of the data on tetralin combustion could not be used, and values of  $c^*$  could not be determined. However, the acceptable data were analyzed using Equation (34). The results showed much scatter and did not seem to correlate with anything, including temperature. In fact, it seemed that the rate of combustion tended to decrease with increased temperature, which is not logical. In an effort to check this result, an experiment was conducted with the specific aim of finding the temperature dependence of the rate of combustion.

A mixture of 99% argon with tetralin and oxygen representing an equivalence ratio of 0.5 was prepared. Experiments were run, repeating the same conditions until the rate of combustion at a particular temperature could be estimated with confidence. Figure 63 shows the results for 9 psia. The temperatures were spread as far as was consistent with obtaining good combustion measurements. If the temperature was too high, there was no measureable ignition delay and often the mixture detonated. If the temperature was too low, the reflected rarefaction shock wave interfered with the combustion. Nevertheless, there appears to be a definite trend of increasing rate with increasing temperature, in spite of considerable scatter in the data. Figure 64 shows the results for 15 psia. The rates of combustion are somewhat higher but show about the same temperature dependence.

With the knowledge that the proper temperature dependence is found (increasing rates with increasing temperature) and that pressure has a beneficial effect on the combustion rate, the tetralin data were re-examined. There are too few points at each set of conditions to analyze the results statistically, but it

Figures 63 and 64 follow

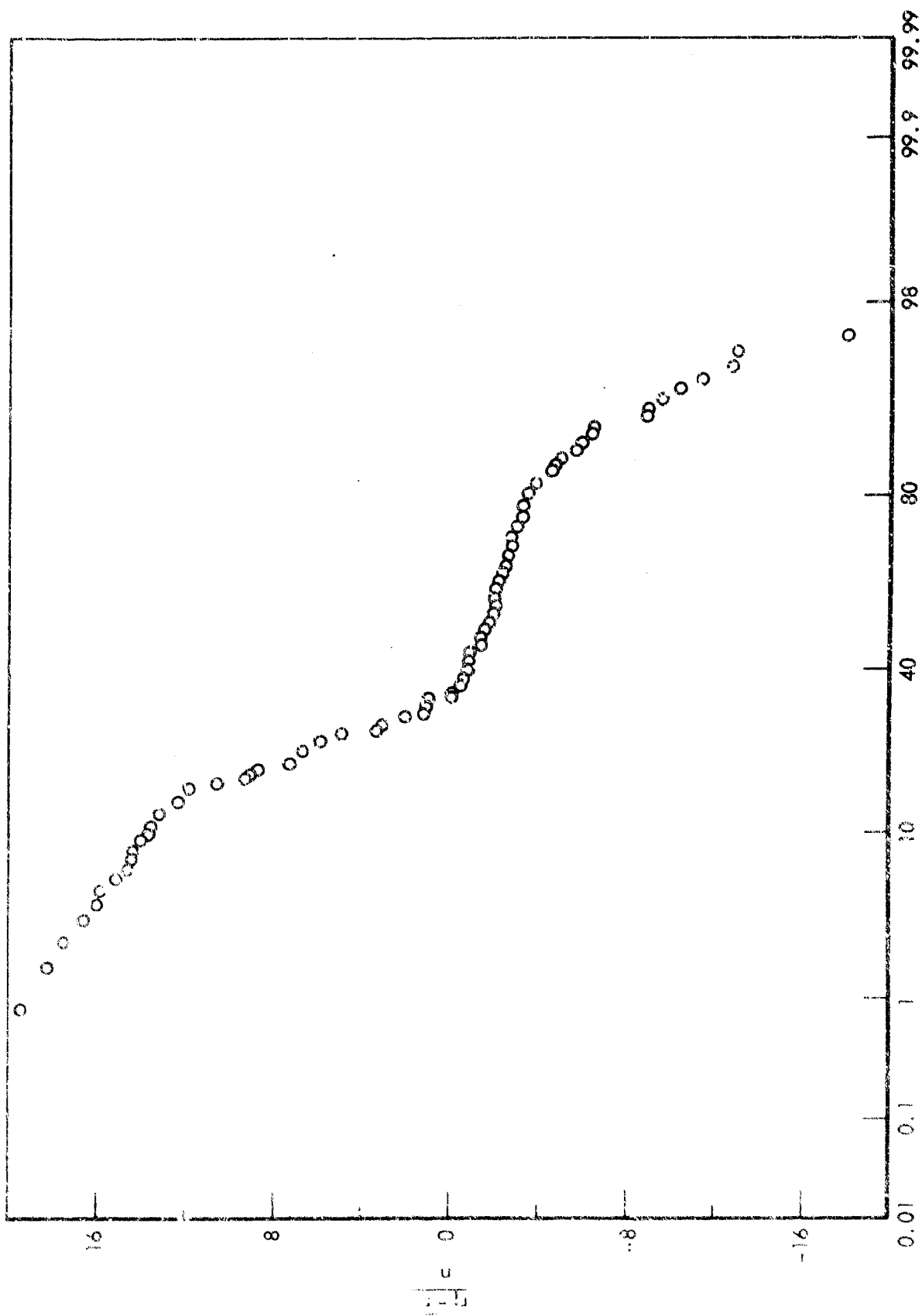


Figure 50. PROBABILITY PLOT OF TETRALIN DATA FOR LOGARITHMIC EQUATION



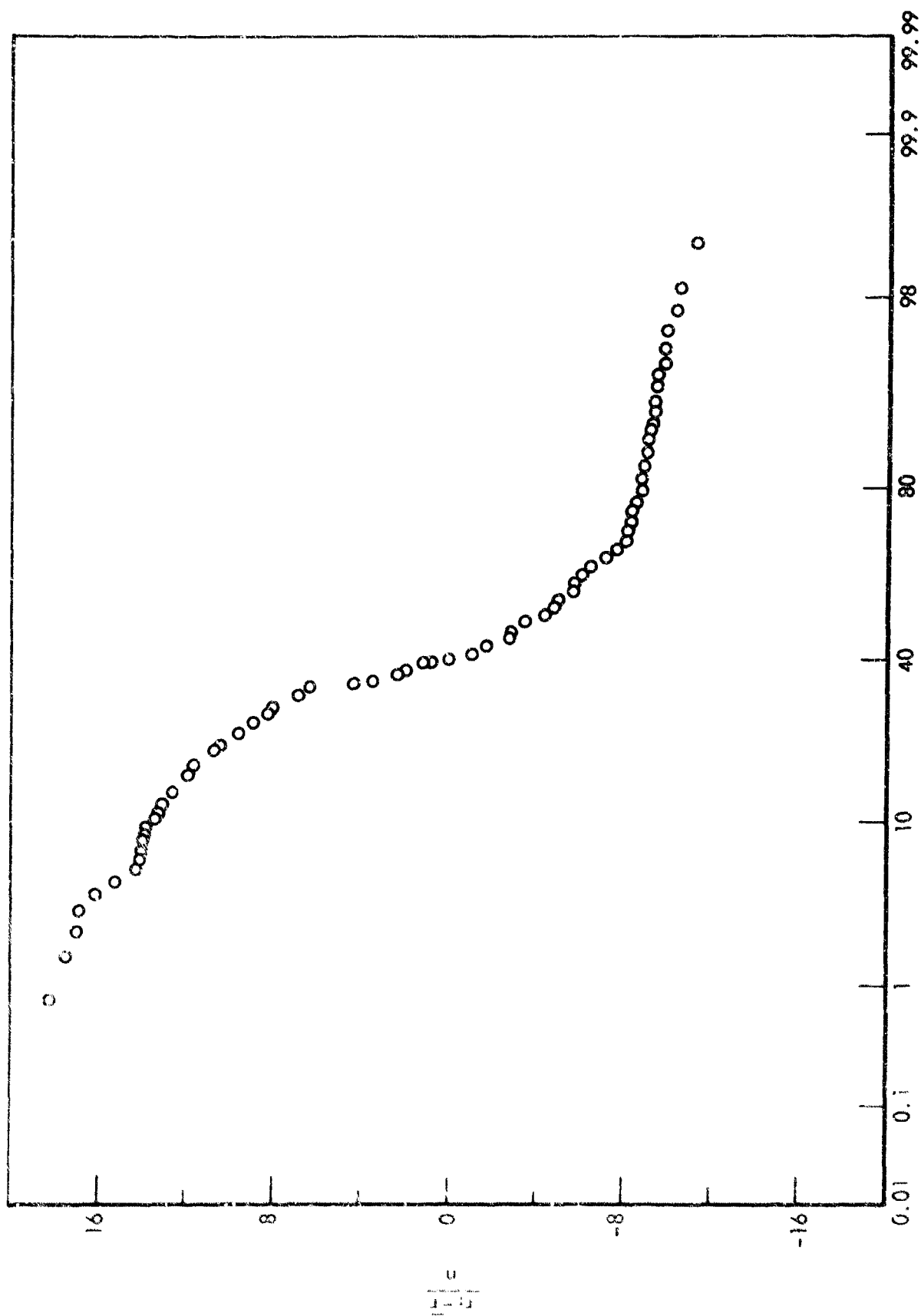


Figure 51. PROBABILITY PLOT OF TETRALIN DATA FOR EXPONENTIAL EQUATION

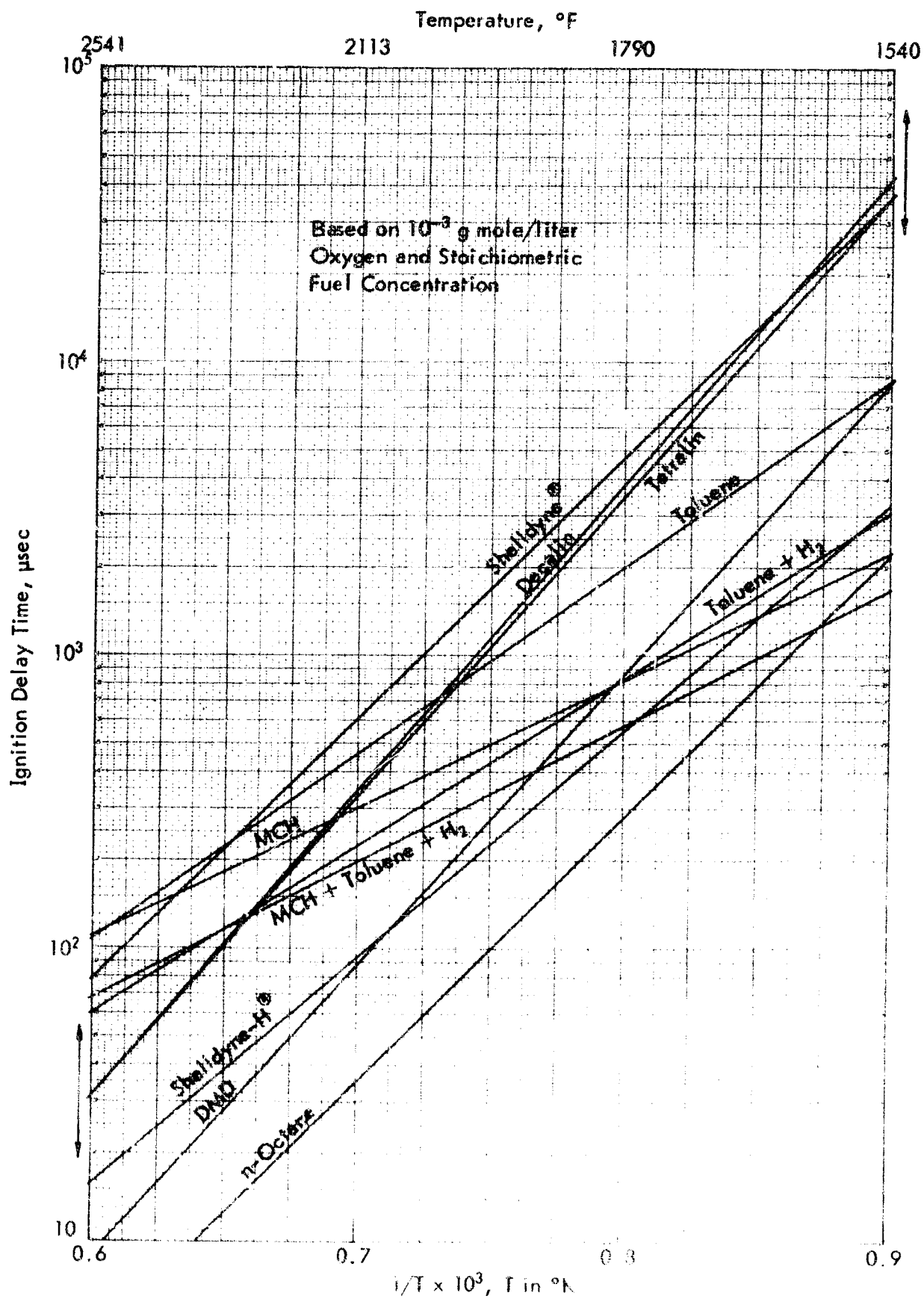


Figure 52. CORRELATIONS OF IGNITION DELAY TIMES

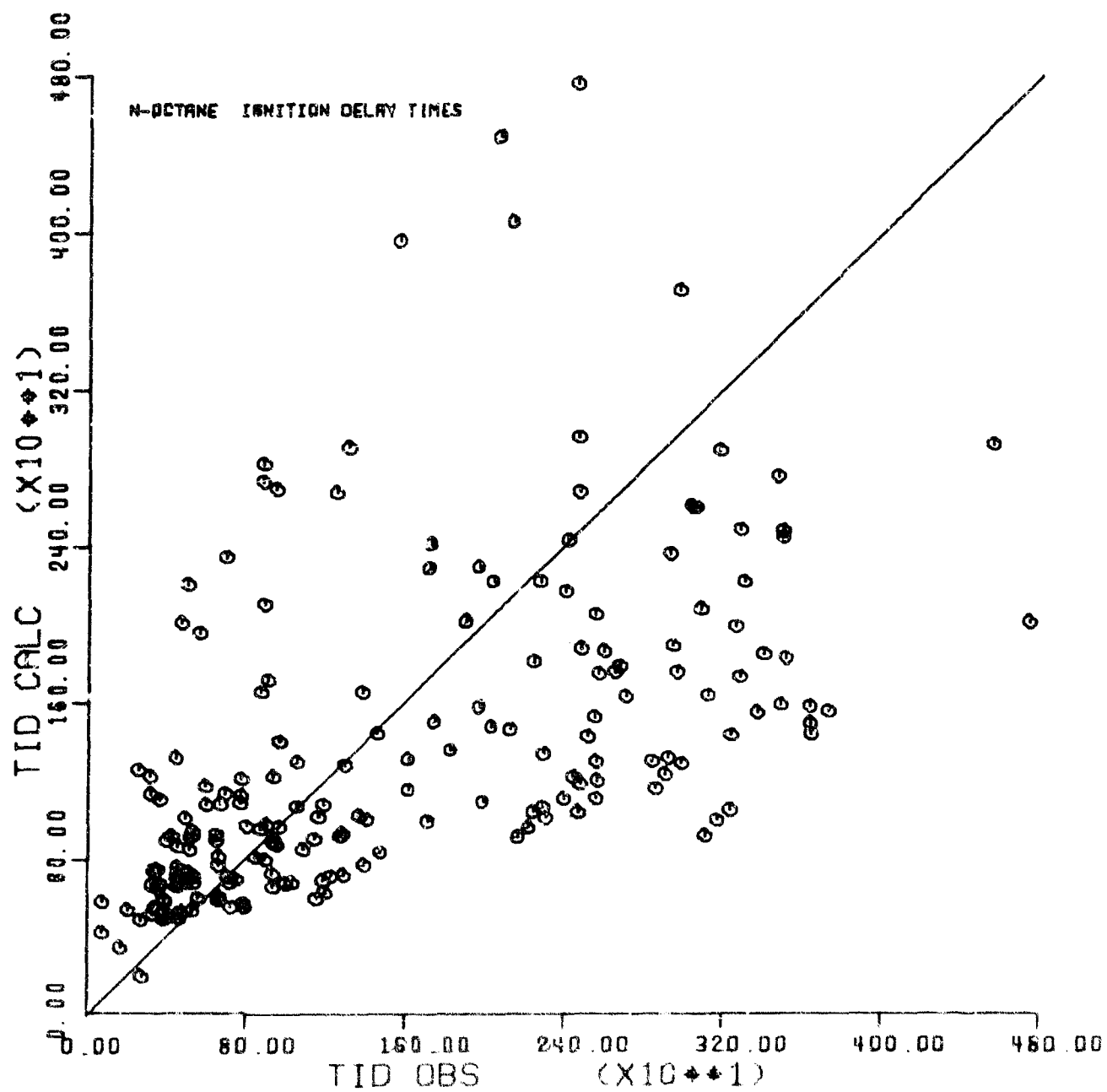


Figure 53. IGNITION DELAY TIMES OF n-OCTANE

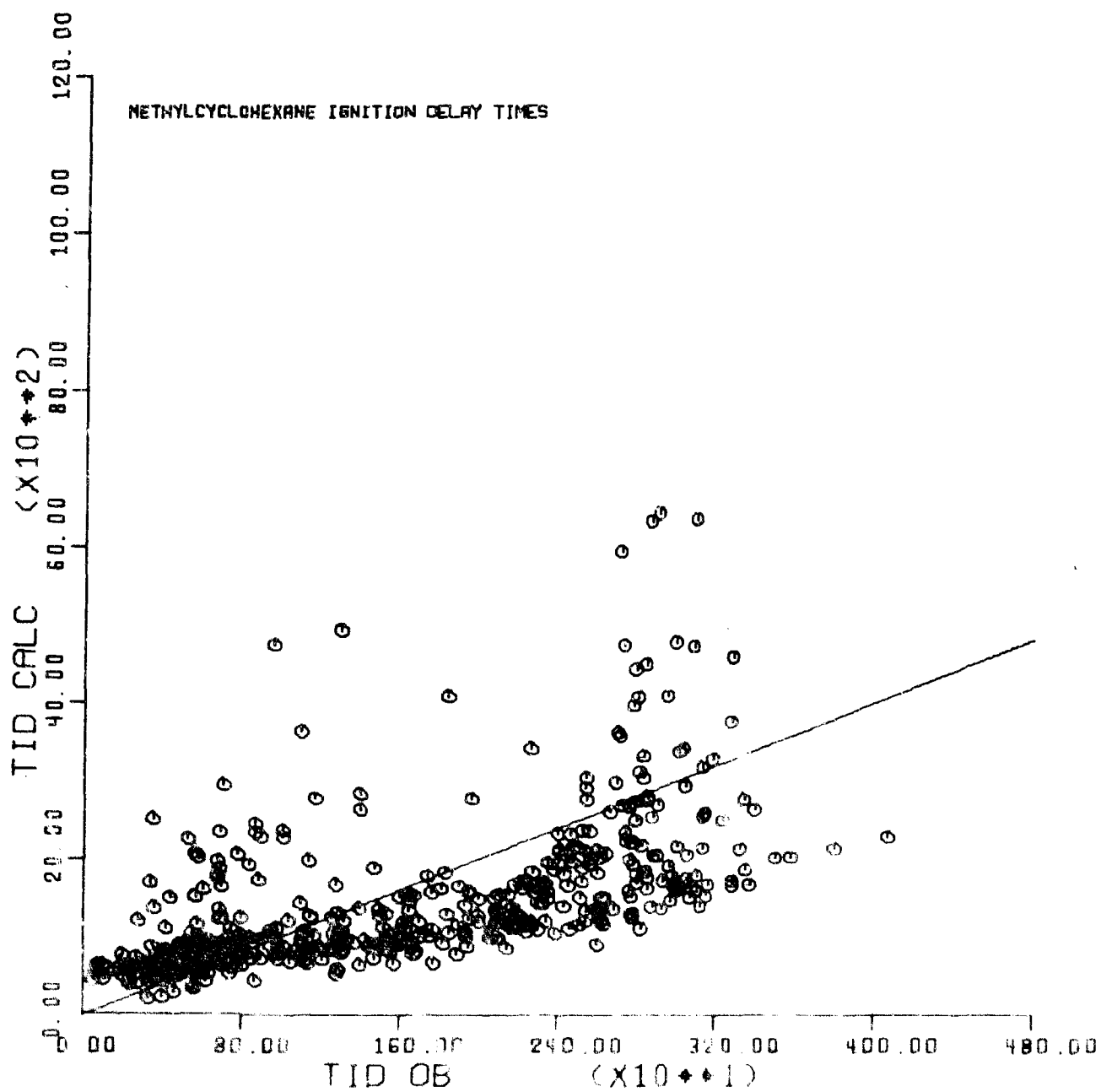


Figure 54. IGNITION DELAY TIMES OF METHYLCYCLOHEXANE

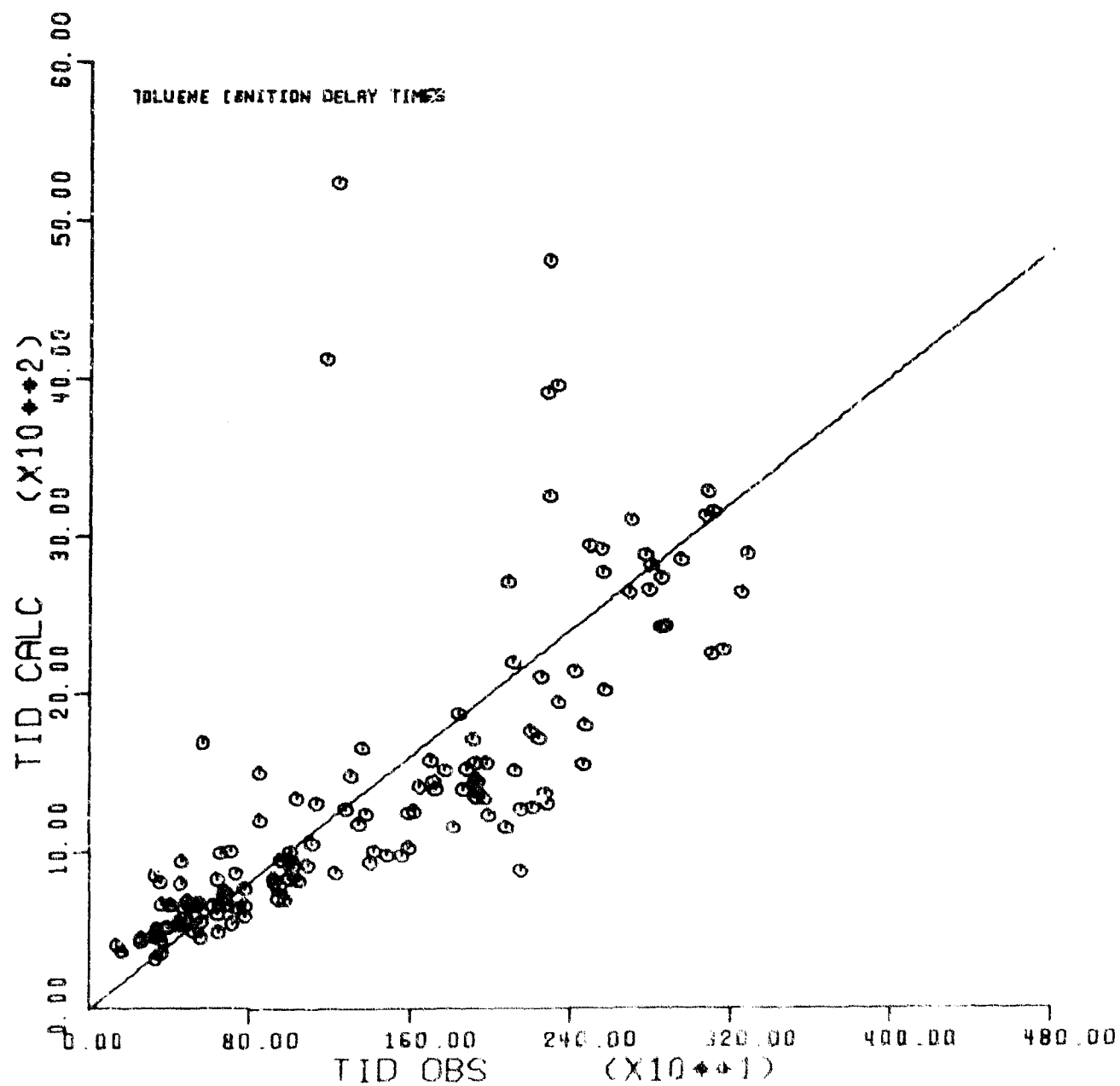


Figure 55. IGNITION DELAY TIMES OF TOLUENE

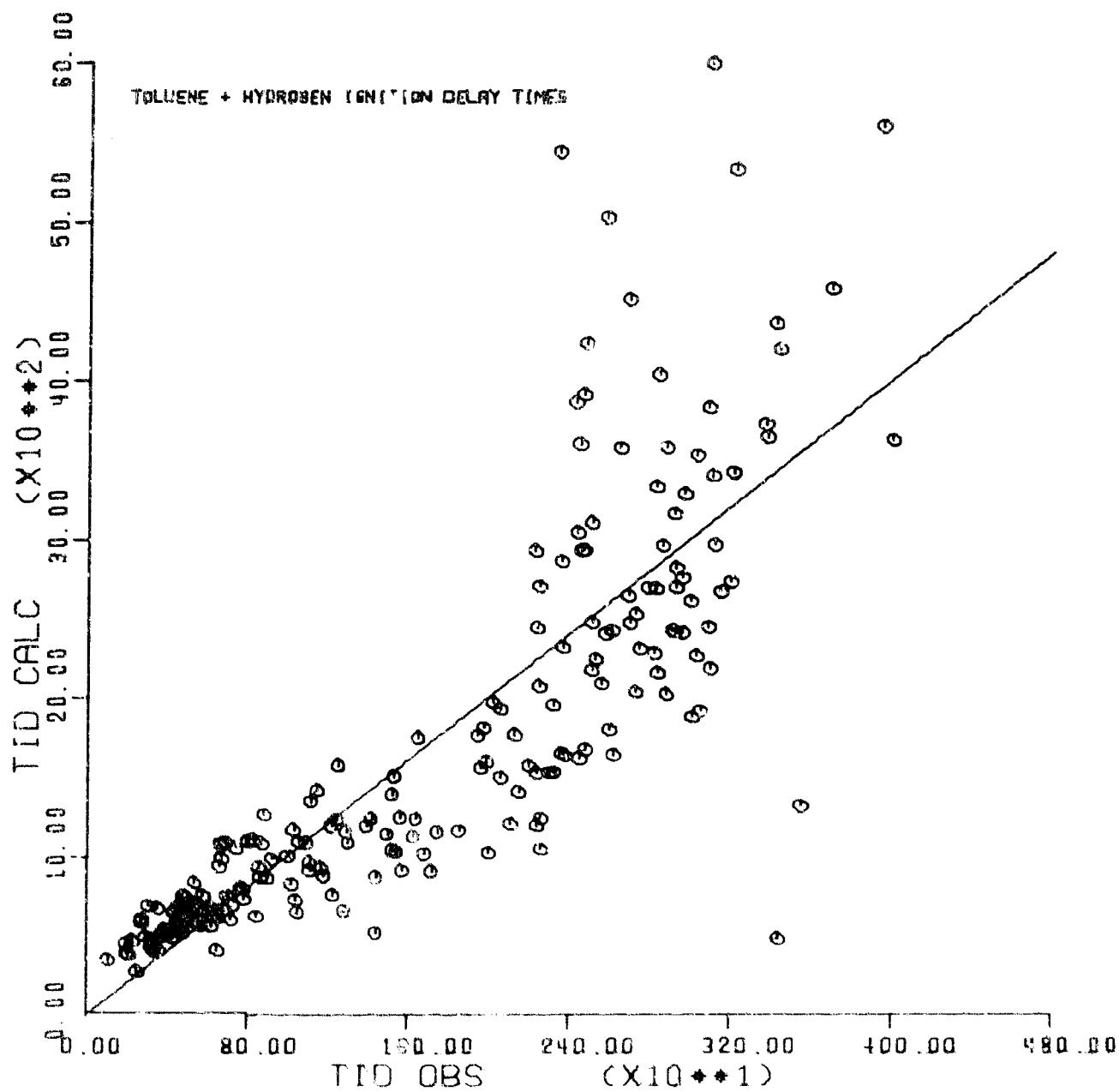


Figure 56. IGNITION DELAY TIMES OF TOLUENE-HYDROGEN MIXTURES

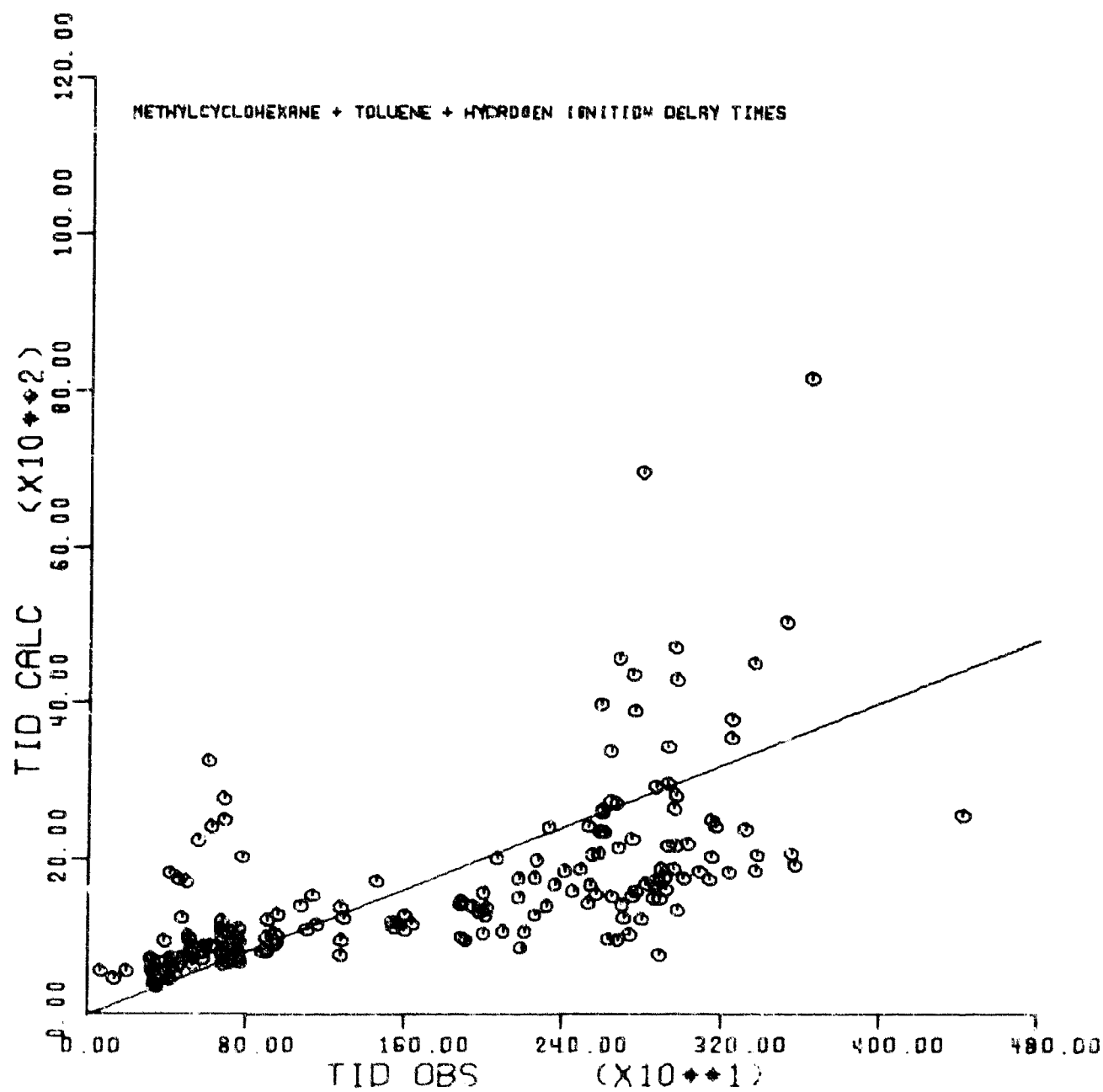


Figure 57. IGNITION DELAY TIMES OF METHYLCYCLOHEXANE-TOLUENE-HYDROGEN MIXTURES

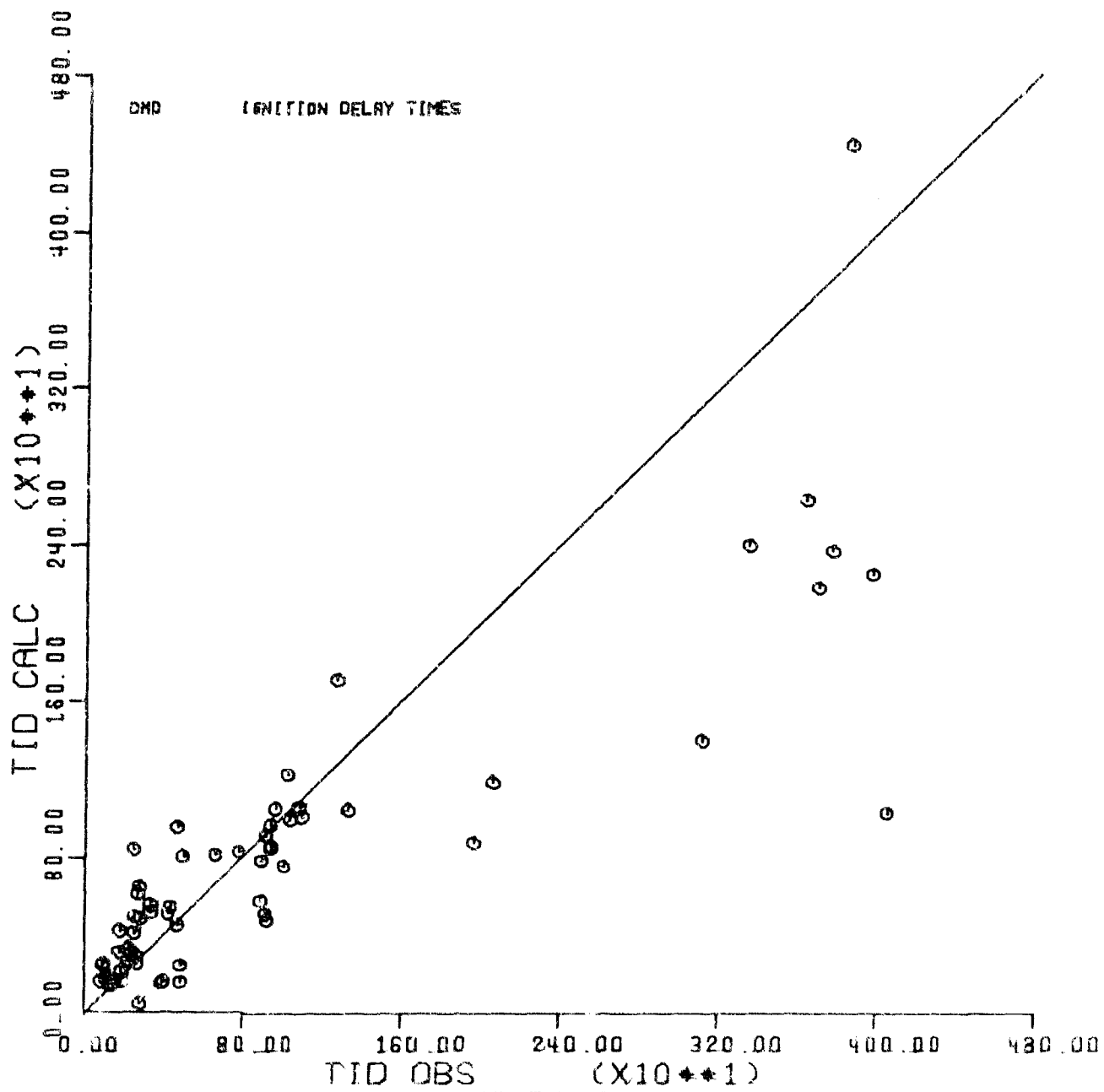


Figure 58. IGNITION DELAY TIMES OF DIMETHANODECALIN



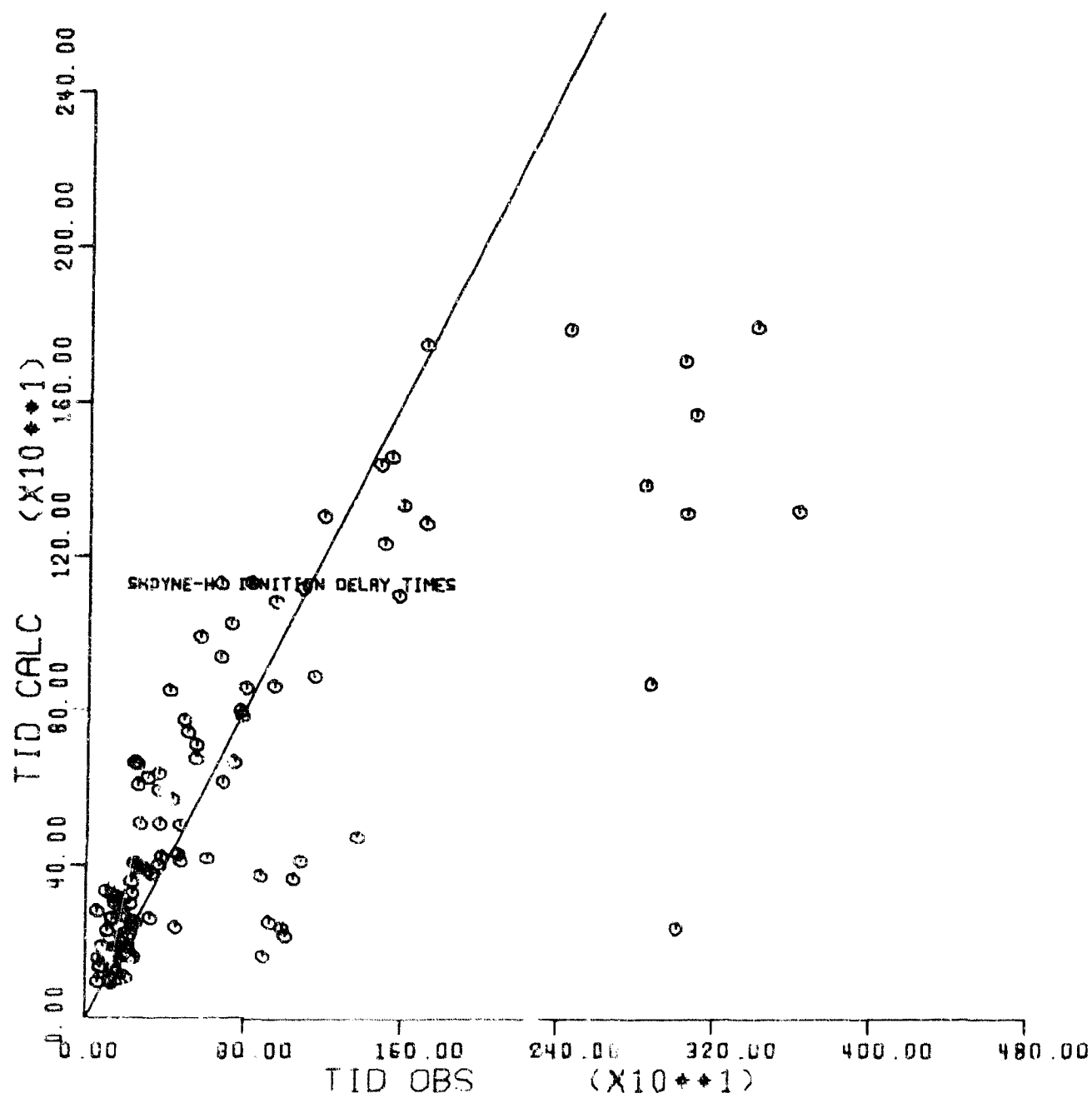


Figure 59. IGNITION DELAY TIMES OF SHELLDYNE-H<sup>®</sup> FUEL

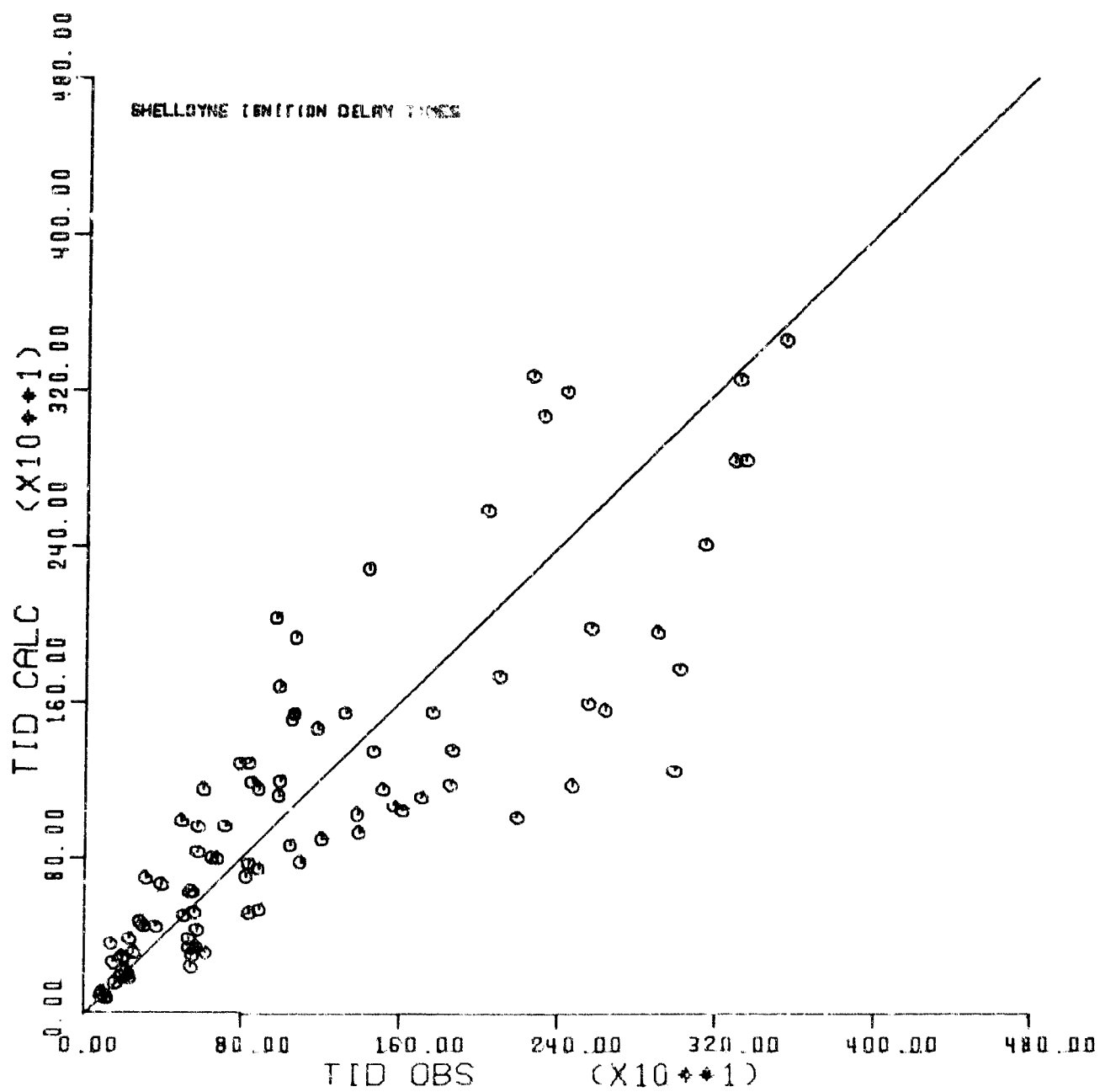


Figure 60. IGNITION DELAY TIMES OF SHELLDYNE<sup>(R)</sup> FUEL

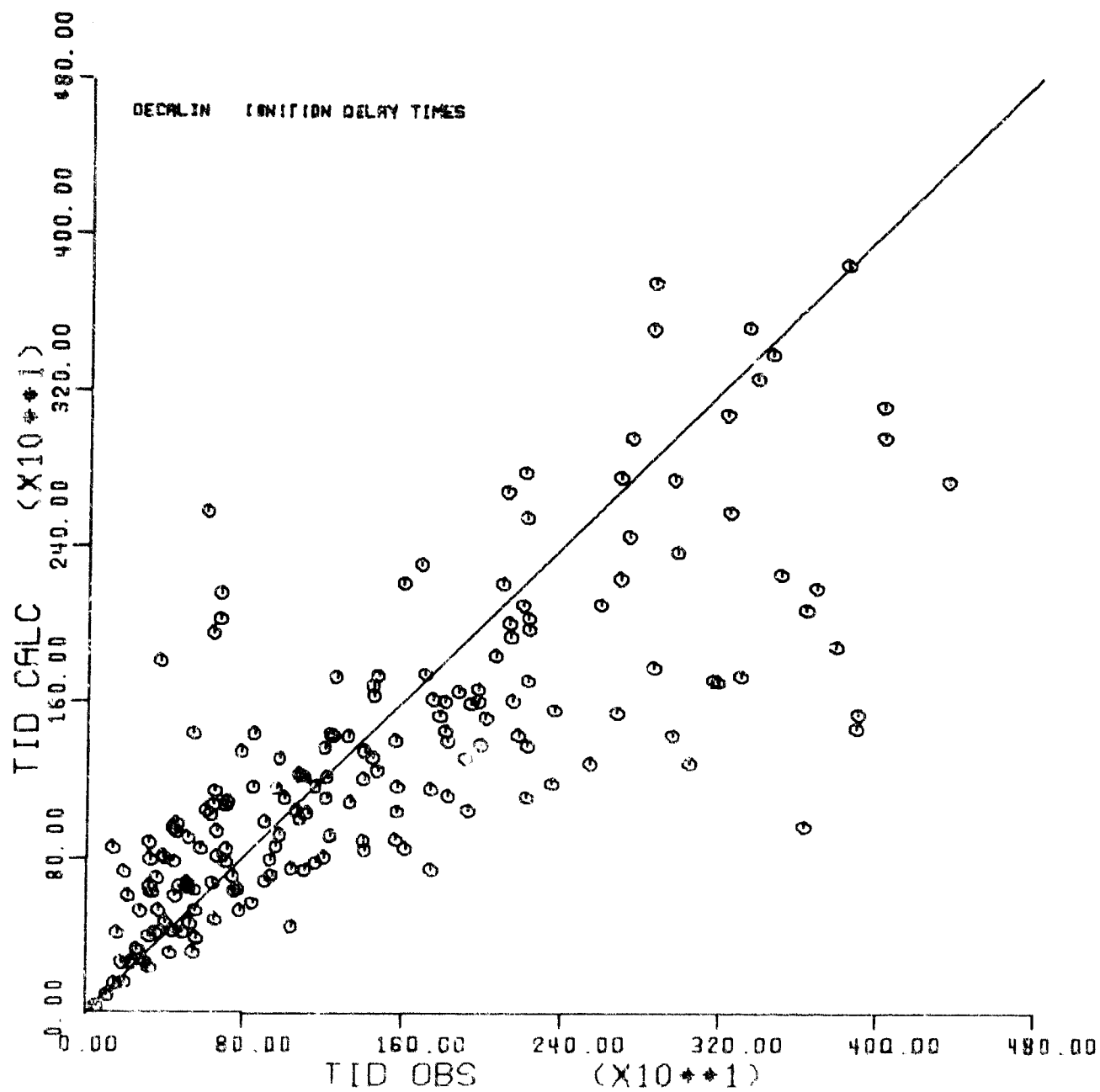


Figure 61. IGNITION DELAY TIMES OF DECALIN

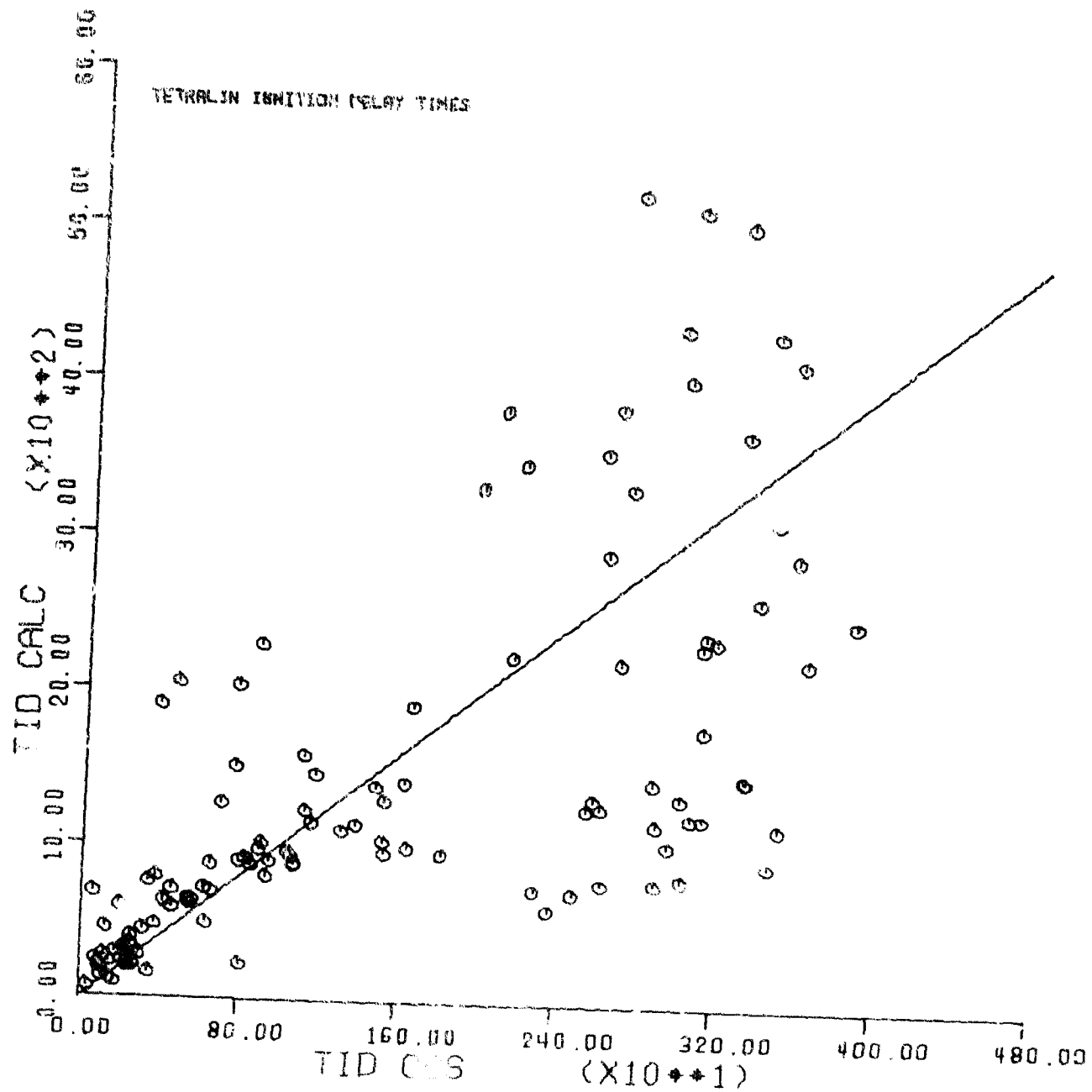


Figure 62. IGNITION DELAY TIMES OF TETRALIN

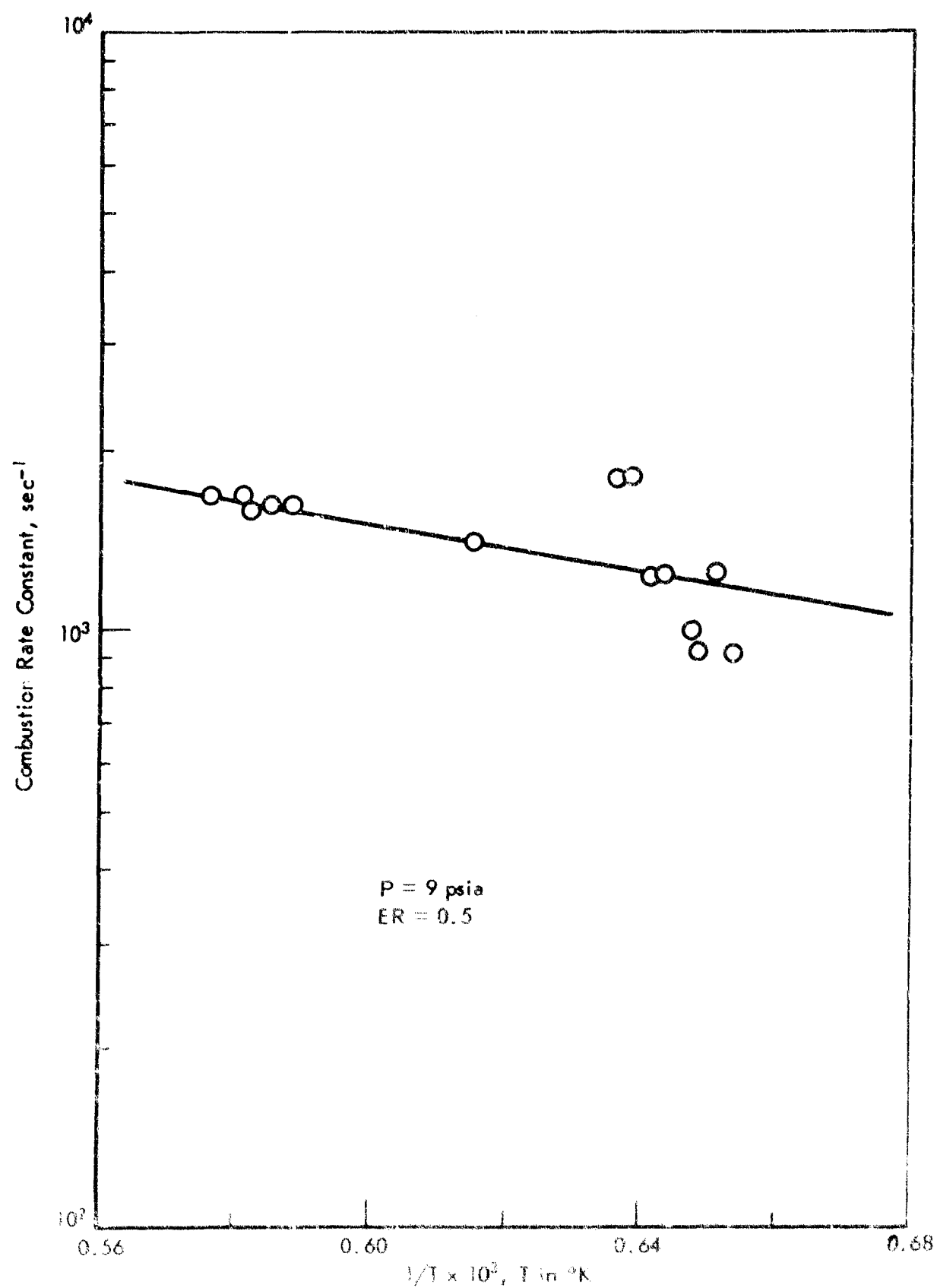


Figure 63. RATE OF COMBUSTION OF TETRALIN

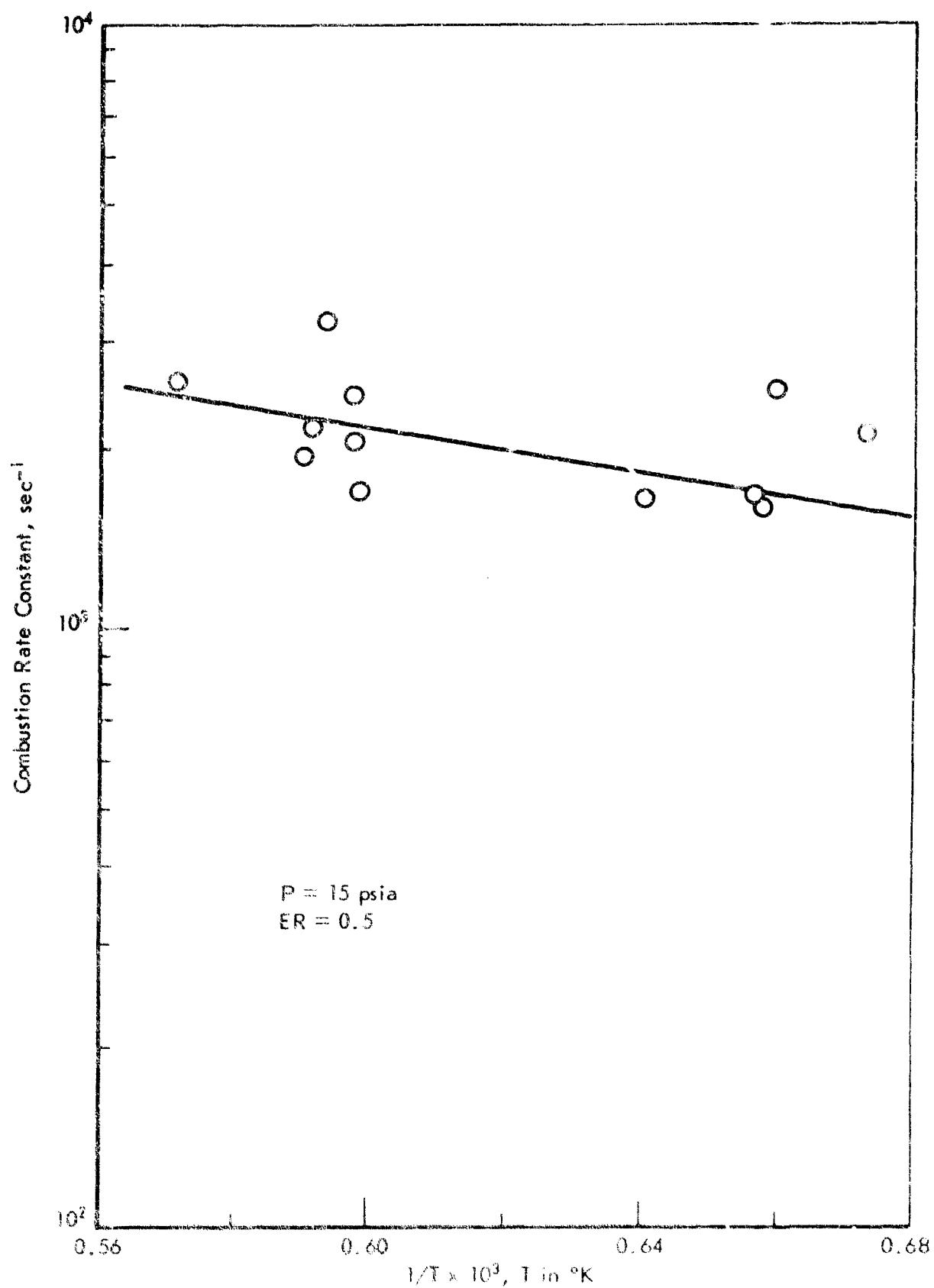


Figure 64. RATE OF COMBUSTION OF TETRALIN

appears that the results correlate fairly well. Figure 65 shows all of the tetralin combustion data, including the new data, with approximate correlating lines. The results show that fuel concentration has a beneficial effect on the rate, since the data for 99% argon with ER = 0.5 are higher than those for 99% argon with ER = 0.1. Also, as the amount of inert gas is decreased (at ER = 0.1) the rate of combustion increases.

This work contrasts somewhat with the previous work done of the combustion of n-octane and SHELLDYNE-H<sup>6</sup> fuel<sup>3</sup>). At that time no effect of fuel-oxygen ratio or pressure was noted. Further work will be done to clarify and mathematically describe the results, and other systems will be investigated.

#### FUTURE PROGRAM

The study of the stability of platinum catalysts for the dehydrogenation of naphthenes is continuing. Future work will include investigations of the effects of metal dispersion and certain metal additives on catalyst stability. The most promising catalysts will be tested in 50 to 100 hour runs at constant reaction conditions. Screening of granular catalysts for dehydrogenation activity will continue on a limited scale as new knowledge indicates any promising compositions.

The search will continue for possible second generation endothermic fuels. Dehydrogenation of bridged-ring naphthenes such as bicyclooctane will yield 1500 to 1800 Btu/lb heat sink when dehydrogenated to bicyclooctatriene. Subsequent double bond isomerization to cyclopropane rings would increase the heat sink to about 2300 Btu/lb. Dehydrocyclization of alkyl naphthenes to the corresponding aromatics, such as 1,2-diethylcyclohexane to naphthalene, would give about 1500 Btu/lb. These studies will be done initially in a pulse reactor. In the near future we expect to examine the possibility of dehydrogenating a highly naphthenic jet fuel and to prepare and test a high molecular weight naphthenic type fuel in the light gas oil range.

We shall continue to search for additives that will enhance the rate of thermal cracking of hydrocarbons. The aim of this study is to reduce both the reaction temperature and the coke made during the cracking reaction.

As a possible method for reducing the pressure drop in the reactor tube, we have been searching for a dispersed-type catalyst. Such a catalyst could be either dissolved in the liquid feed, added as a vapor to the feed vapor, or added as a finely divided solid to the feed vapor. In our previous contract we examined additives that were dissolved in the liquid feed. Some encouraging results were obtained and this study is continuing.

Development will continue on the catalytic wall reactor. The most immediate problem is the measurement of the stability and life of a wall catalyst for dehydrogenation of naphthenes. Catalyst formulations which were found to be most promising in activity screening studies will be used in preparing catalytic coatings for longer stability tests in the bench-scale reactor. If results are favorable, tests will be made in the Fuel System Simulation Test Rig to assess fully the capability of wall catalysts for dehydrogenating naphthenes. These tests will

Figure 65 follows

be made in the Fuel System Simulation Test Rig to assess fully the capability of wall catalysts for dehydrogenating naphthenes. These tests will be of sufficient duration to determine catalyst activity and stability under various conditions. Concurrently, the search will continue for wall catalysts with even more improved mechanical properties, activity, and stability.

The mathematical model of the Catalytic Wall Reactor will be improved. The current model, which includes a kinetic model for methylcyclohexane dehydrogenation, will be used to design the experiments for the Fuel System Simulation Test Rig so as to obtain data at an optimum set of conditions. These data will help to improve the mathematical model. The reaction model can be used to study the effects of geometry, flow rate, heat flux, and fuel conditions on the performance of a wall catalyst. After further development of the reactor model, it will be possible to calculate the upper limits of heat flux, conversion, and temperature of each reaction system as determined by the stability and activity of the catalyst and fuel of that system.

A Catalytic Continuous Stirred Tank Reactor has been designed for use in measuring reaction kinetics and catalyst stability. Equipment modification will begin soon and the reactor will be available for experiments in a short time. The reactor will be used to measure the intrinsic kinetics of dehydrogenation reactions on the best catalyst candidates. Data will be used to formulate kinetic models for the reaction systems. Dehydrogenation of MCH will be studied first to check the reaction kinetics that are currently used. Later experiments will be run to determine an acceptable model for the decalin dehydrogenation system.

Further calibration and evaluation of the beta-ray backscatter instrument on tubes of different dimensions and on tubes made of other alloys will be made. Now that the beta-ray backscatter instrument is available for rating tubes, the Catalyst and Fuel System Test Rig will be used in evaluating the deposits formed by fuels in various physical and chemical states: liquid, gas, vaporizing mixture, reacting mixture, and product mixture. Studies will continue on the use of additives to enhance the thermal and storage stability of fuels. Tests on thermal stability will be conducted in the recently modified JFTOT in order to determine if results from a one-cycle test such as this are better than measurements from a recycle test in the SD/M-7 coker.

Combustion studies on the shock tube will continue with the decalin dehydrogenation system. Ignition delay times will be measured for mixtures of components from the system: decalin, tetralin, naphthalene, and hydrogen, in proportions representative of products from the dehydrogenation reaction. Further experiments will be made on the combustion of tetralin and other fuels to define more accurately the relation between the combustion rate, pressure, oxygen concentration, and fuel concentration.

#### RELATED DEVELOPMENTS AND APPLICATIONS

An interesting paper was presented by D. C. Thomas and P. H. Hayes<sup>26)</sup> entitled "High Performance Heat Transfer Surfaces." They showed that heat transfer coefficients through tube walls could be increased by combining rectangular fins on the outside with twisted tape or coiled wire on the inside. Although the



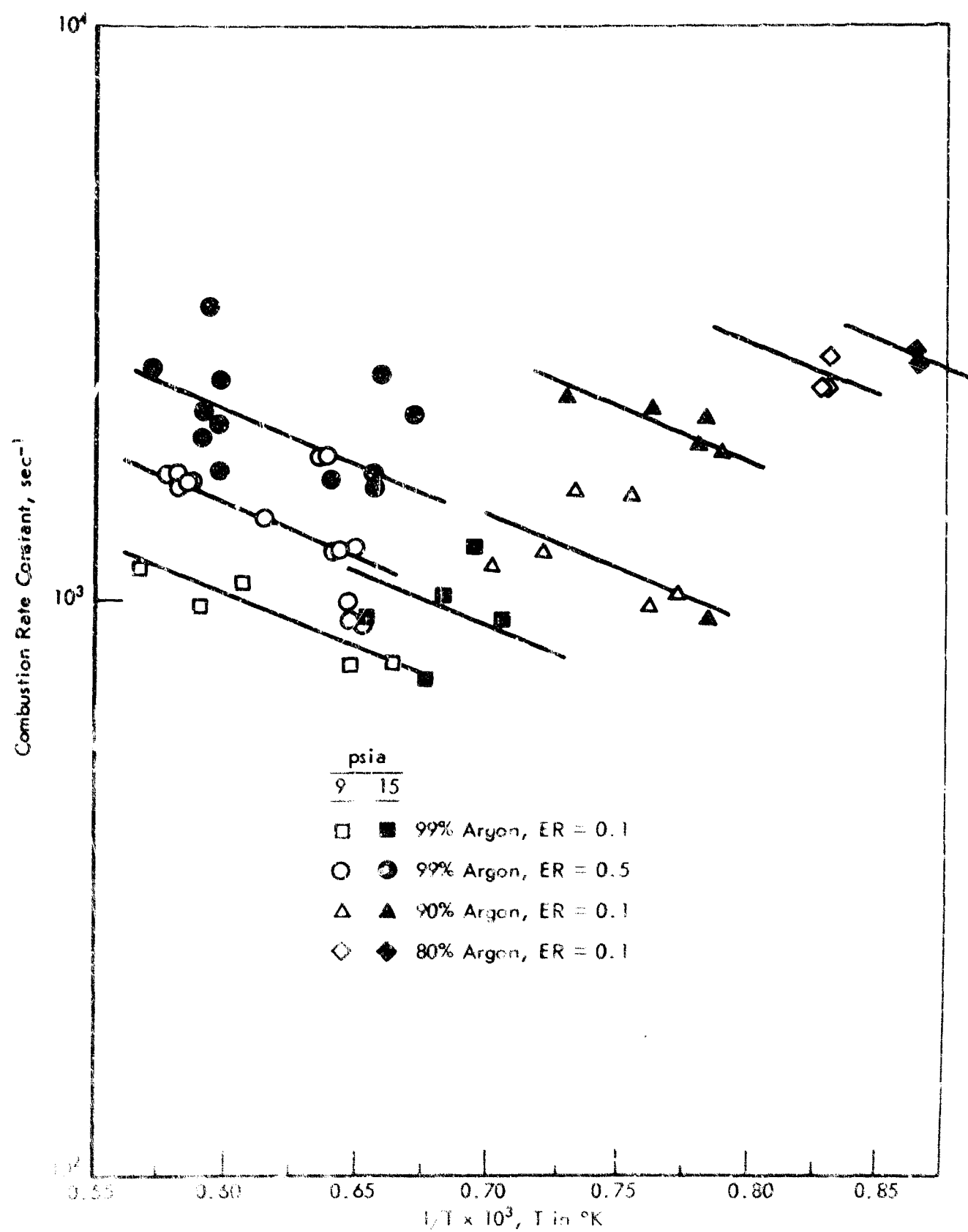


Figure 65. COMBUSTION OF TETRALIN

system studied involved condensing or evaporating water, the results showed benefits in heat transfer coefficients from two- to ten-fold and should be applicable to reacting systems with suitable modifications.

An article by Watkins and Jacobs<sup>27)</sup> details the use of hydrocracking to upgrade vacuum bottoms, low quality straight run, and catalytically cracked kerosene to commercial jet fuel specifications. Although the article applies only to the manufacture of commercial jet fuel, this method could be applied to selected petroleum feed stocks to produce naphthene concentrates which might serve as cheap endothermic fuels.

An article of interest by Whisman and Ward<sup>28)</sup> on the storage stability of high temperature fuels showed by means of fuel components labelled by C<sup>14</sup> that the contribution to coker deposits in different thermally stable jet fuels was highest from aromatic compounds containing a C<sub>5</sub>-ring, but the effect of any component was strongly dependent on the gross hydrocarbon environment. It also showed that an amine type antioxidant formed more filterable deposits in a high temperature environment than did a phenolic type.

An important paper delineating the problems inherent in extending flight speeds up to Mach 6 appeared in Aeronautics and Astronautics by M. P. Dunnam and H. I. Bush.<sup>29)</sup> The authors outlined the "broad range of technical advances which will enable turbomachinery to power demanding new missions and will pave the way for exotic new engines that may supplant rockets almost up to orbital speeds." An accompanying article by W. E. Lanar<sup>30)</sup> is also valuable.

Efforts to develop high temperature materials for advanced gas-turbine engines was reported by John C. Freche and Robert W. Hall<sup>31)</sup> of Lewis Research Center, NASA. "NASA Programs for Development of High Temperature Alloys for Advanced Engines" dealt chiefly with work on the development of nickel- and cobalt-base alloys, chromium-base alloys, materials strengthened by dispersion, composite materials, and protective coatings. The development of an advanced cast nickel-base alloy, NASA-TRW VI-A, was particularly significant. This was reported to have a high temperature life of 1000 hr at 1890°F.

Another interesting paper was "The Active Cooling of a Hydrogen Fueled Scram-Jet Engine" by L. L. Pagel and W. R. Warmbold<sup>32)</sup>, McDonnell-Douglas Company, St. Louis. The results of this study indicate that Mach 12 flight at equivalence ratios of less than 1 can be achieved with a regenerative system using the hydrogen fuel as coolant in heat exchangers constructed from super alloys. Heat exchanger designs were based on TD Nickel-chromium construction. The authors suggest that this is a satisfactory material for use as a refractory metal in heat exchangers and that the use of ceramic coatings appears less urgent.

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Tables 55 and 56 follow

### Description of the Pulse Reactor.

The pulse reactor was a 1/4-in. OD tube of stainless steel type 304 with 9-1/4 in. length and 0.035 in. wall thickness. Swagelok Tees were fastened at each end and one arm of the Tee served as an injection port. A rubber septum (GLC type) was held in place by the fitting nut and the feed was injected through this septum from a syringe. A 5-in. length of the reactor tube was surrounded by a secondary furnace liner and the whole was heated by an electric furnace. The secondary liner had seven radial drilled holes for thermocouples, and the holes were located as shown in Figure 66. A schematic diagram of the pulse reactor is shown in Figure 67.

All lines were 1/4-in. OD tubing of stainless steel type 304. About 28 in. of line just prior to the reactor was wrapped with heating tape and constituted a gas preheater. About 8 in. of the preheater section was filled with quartz chips (10-20 mesh size).

In the pulse reactor system the carrier gas was metered through a rotameter (Figure 67) and passed through the preheater section into the reactor. The exit gas passed into a manifold and then into the GLC. The purpose of the manifold was to maintain the exit gas pressure slightly greater than the gas pressure in the GLC. This was done by adjusting the pressure control valve and the vent valve. The manifold was wrapped with heating tape and was maintained at 302° to 356°F. The injection port temperature was about 450°F. The pressure control and the vent valves were needle valves (Hoke No. 1315) and the GLC valve was a lever operated valve (Hoke No. 490).

To carry out an experiment the reactor was brought to temperature, and the carrier gas flow rate, reactor pressure and manifold pressure were adjusted by means of the appropriate flow control valves. Then with inert gas flowing to the GLC a pulse was injected through the lower injection port and subsequently analyzed. This gave an analysis of the starting material. A pulse was then injected in the top injection port, passed over the catalyst, and analyzed.

In this system the space velocity was obtained from the inert gas flow rate. Figure 68 shows the pulse reactor system with the secondary furnace liner in place.

Figures 66, 67 and 68 follow

Run No.: 11623-  
 Catalyst No.: 100  
 Temperature, °F: 100  
 Block: 100  
 Wall: 100  
 Catalyst Bed: 100  
 Profile: 100

Run No. 11623-	171	172	173	174	175	176	177	178-1	178-2	179	180-1	180-2	181	182	183	184	185	186
Catalyst No.	1000-104				1000-105				1000-106				1000-107				1000-108	
Temperature, °F	100				100				100				100				100	
Block	100				100				100				100				100	
Wall	100				100				100				100				100	
Catalyst Bed	100				100				100				100				100	
Profile	100				100				100				100				100	
Product Analysis, %w	100				100				100				100				100	
Cracked, liq.	100				100				100				100				100	
trans DHN	100				100				100				100				100	
cis DHN	100				100				100				100				100	
THN	100				100				100				100				100	
N	100				100				100				100				100	
Others	100				100				100				100				100	
DHN Conversion, %w	100				100				100				100				100	
Selectivity for THN + N, %w	100				100				100				100				100	
ΔT, °F	100				100				100				100				100	

- a) cis to trans isomerization during the run  
 b) two components  
 c) four components  
 d) appeared to be more than one component  
 e) eight components  
 f) two components that emerged after cis DHN and one component that emerged after trans DHN
- h) emerged after cis DHN  
 m) catalyst almost completely deactivated at  
 n) emerged after trans DHN  
 p) twelve components  
 q) one component emerged after trans DHN (7.0%)  
 r) five components

Run No. 11623-	16	17-1	17-2	18	20	21-1	21-2	22	23	24	25	26-1	26-2	27
Catalyst No.	10860-137B				10860-138B				10860-139B				10860-140B	
Temperature, °F	100				100				100				100	
Block	100				100				100				100	
Wall	100				100				100				100	
Catalyst Bed	100				100				100				100	
Profile	100				100				100				100	
Product Analysis, %w	100				100				100				100	
Cracked, liq.	100				100				100				100	
trans DHN	100				100				100				100	
cis DHN	100				100				100				100	
THN	100				100				100				100	
N	100				100				100				100	
Others	100				100				100				100	
DHN Conversion, %w	100				100				100				100	
Selectivity for THN + N, %w	100				100				100				100	
ΔT, °F	100				100				100				100	

- a) cis to trans isomerization during the run  
 b) reaction time 15 minutes  
 c) one component emerged after trans DHN (1.9%) and one component after cis DHN (0.2%)  
 d) eight components  
 e) one component emerged after trans DHN (4.8%) and one after cis DHN (9.5%)  
 f) reaction time 15 minutes  
 g) four components
- h) reaction time 10 minutes  
 m) five components  
 n) emerged after trans DHN  
 p) ten components  
 q) emerged after cis DHN  
 r) six components  
 s) one component emerged after trans DHN (0.7%) and

Run No. 11623-	46	47	48-1	48-2	49	51	52	53-1	53-2	54	56-1	56-2	57	58
Catalyst No.	10860-146				10860-147				10860-148				10860-149	
Temperature, °F	100				100				100				100	
Block	100				100				100				100	
Wall	100				100				100				100	
Catalyst Bed	100				100				100				100	
Profile	100				100				100				100	
Product Analysis, %w	100				100				100				100	
Cracked, liq.	100				100				100				100	
trans DHN	100				100				100				100	
cis DHN	100				100				100				100	
THN	100				100				100				100	
N	100				100				100				100	
Others	100				100				100				100	
DHN Conversion, %w	100				100				100				100	
Selectivity for THN + N, %w	100				100				100				100	
ΔT, °F	100				100				100				100	

- a) cis to trans isomerization during the run  
 b) seven components  
 c) reaction time 10 minutes  
 d) six components  
 e) four components  
 f) emerged after THN





Table 55 (Contd). DEHYDROGENATION

Run No. 11355-	146	147	146-1	148-2	149	99	109	101	105	106	107	108	105	104	111
Catalyst No.	9874-110B					9874-180A					9874-187A				
Temperature, °F	842	932	1022	1112	1202	842	932	1022	842	932	1022	842	932	1022	842
Block	704-07	759-65	824-30	905-18	1015-1175	725-29	788-99	858-997	727-32	790-813	873-1011	736-41	799-820	878-1008	761-828
Wall	617-32	657-78	698-73	744-83	862-1156	650-67	707-81	806-995	669-89	723-824	837-997	667-82	720-810	844-977	770-817
Catalyst Bed	603-09	635-44	671-80	712-34	815-1130	632-40	675-94	723-984	6114-55	657-723	752-997	634-51	684-723	750-935	691-815
Profile	609-15	644-50	682-89	734-38	835-1017	637-44	680-87	725-905	648-53	689-700	740-997	649-35	691-702	743-992	658-799
	626-30	664-68	711-14	774-72	1015-1175	651-55	696-700	745-797	657-60	702-05	750-987	662-64	707-09	754-988	656-763
Product Analysis, %															
Cracked, liq	0.0	0.0	0.1	0.6	4.2	0.0	0.0	0.2	0.0	0.0	0.1	0.0	0.0	0.3	0.3 <sup>a</sup>
trans DHN	30.5 <sup>a</sup>	27.7 <sup>a</sup>	23.5	18.8	15.2	29.0 <sup>a</sup>	26.3 <sup>a</sup>	22.2	26.6 <sup>a</sup>	25.6	22.5	26.4 <sup>a</sup>	24.6	22.5	28.5 <sup>a</sup>
cis DHN	29.4	24.8	20.3	16.5	27.1	40.9	37.0	40.4	45.2	42.0	50.5	45.9	41.6	51.0	52.1
THN	17.9	14.2	9.3	5.1	5.2	9.9	7.7	6.5	8.2	6.1	5.1	7.8	6.0	4.4	5.3
N	21.8	33.3	46.8	56.9	46.9	20.1 <sup>b</sup>	28.8	28.9	20.0	26.3	19.4	19.9	27.8	15.0 <sup>c</sup>	11.0
Others	0.0	0.0	0.0	0.1 <sup>b</sup>	1.5 <sup>c</sup>	0.1 <sup>b</sup>	0.2 <sup>b</sup>	1.8 <sup>d</sup>	0.0	0.0	2.8 <sup>e</sup>	0.0	0.0	3.9 <sup>f</sup>	3.0 <sup>g</sup>
DHN Conversion, %	39.3	47.1	55.8	62.6	57.4	29.7	36.3	37.0	27.8	32.0	26.6	27.3	33.4	23.2	19.2
Selectivity for THN+N, %	100	100	99.8	98.4	90.1	99.3	99.4	97.0	100	100	90.6	100	100	81.9	82.8
ΔT, °F	14	22	45	133	389	20	74	261	20	101	257	16	92	248	140

a) cis to trans isomerization during run

b) emerged after cis DHN

c) one component emerged after trans DHN (0.7%), and one after cis DHN (0.7%)

d) one component emerged after trans DHN (0.9%), and one after cis DHN (0.9%)

e) one component emerged after trans DHN (1.0%), and one after cis DHN (1.4%)

f) one component emerged after trans DHN (1.5%), and one after cis DHN (1.5%)

g) emerged after cis and trans DHN

Run No. 11325-	138	139	140-1	140-2	141	118	119	120	126	127	128-1	128-2	129	130	131
Catalyst No.	10280-15A					10280-50A					10280-77A				
Temperature, °F	842	932	1022	1112	1202	842	932	1022	842	932	1022	1112	1202	842	932
Block	736-41	799-802	874-78	948-46	1044-49	802-26	885-920	972-1008	711-14	766-70	824-26	891-94	977-1035	640-42	680
Wall	666-82	711-34	759-84	806-42	903-1010	797-802	873-900	950-88	617-28	650-64	687-109	770-840	892-1121	630-42	662-76
Catalyst Bed	635-42	669-78	711-16	748-52	822-44	770-808	838-902	918-88	604-17	635-40	667-75	716-22	770-73	621-26	651-55
Profile	635	669-71	711-08	750-43	822-17	686-812	806-903	894-988	615-26	646-57	682-86	732-29	797-804	628-32	662
	644	684	730-25	748-46	1044-49	802-26	788-903	889-980	633-42	671-74	714-16	770-65	853-33	640-42	680
Product Analysis, %															
Cracked, liq	0.0	0.0	0.0	0.4	1.9	1.5	1.5	2.5	0.0	0.0	0.0	0.9	3.2	0.7	0.3
trans DHN	29.9 <sup>a</sup>	26.4 <sup>a</sup>	21.8	16.4	11.2	27.0 <sup>a</sup>	25.6 <sup>a</sup>	26.8 <sup>a</sup>	34.5 <sup>a</sup>	30.8 <sup>a</sup>	25.3	18.3	12.7	34.5 <sup>a</sup>	31.0 <sup>a</sup>
cis DHN	59.1	35.0	29.6	24.3	21.5	63.0	67.7	63.0	26.5	21.5	17.3	17.0	14.1	27.4	21.6
THN	11.0	9.2	8.1	7.2	5.6	2.1	1.7	1.8	17.5	14.3	10.5	6.7	4.1	14.6	11.5
N	20.0	29.5	40.5	51.4 <sup>b</sup>	59.4 <sup>c</sup>	1.7	0.9	1.0	21.3	33.5	46.9	57.2	64.7 <sup>e</sup>	23.4	35.5
Others	0.0	0.0	0.0	0.2 <sup>b</sup>	0.4 <sup>c</sup>	4.7 <sup>d</sup>	1.6 <sup>d</sup>	4.9 <sup>d</sup>	0.0	0.0	0.0	0.1 <sup>b</sup>	1.2 <sup>e</sup>	0.0	0.0
DHN Conversion, %	30.6	38.3	48.2	59.3	66.9	9.6	5.7	10.2	38.1	47.2	57.0	64.3	72.8	37.6	47.0
Selectivity for THN+N, %	100	100	100	98.1	96.6	35.4	40.4	25.5	100	100	100	98.8	94.0	99.7	99.4
ΔT, °F	16	23	25	36	107	126	--	--	13	14	22	170	229	13	14

a) cis to trans isomerization during run

b) emerged after naphthalene

c) two components - one emerged after trans DHN and one after cis DHN

d) emerged after cis DHN

e) three components, one of which emerged after trans DHN, cis DHN and naphthalene respectively

f) two components that emerged after cis DHN and naphthalene respectively

# HYDROGENATION OF DECALIN OVER VARIOUS CATALYSTS

103	104	111	112	115	96	97	98	99	94	95	142	143	144-1	144-2	108	109	110	114	115	116-1	116-2	
9874-189A		9874-189B			9874-192A					9874-194A		9874-195A		9874-196B		10280-113		10280-113				
932 709-820 718-817 684-721 691-702 (07-99)	1322 878-1000 771-817 750-795 743-982 758-968	842 761-828 858-937 691-815 658-749 656-765	1322 826-916 858-937 754-905 696-887 696-852	1322 842-916 858-937 754-905 696-887 788-962	842 750-86 687-78 639-48 644-48 653	932 797-850 707-864 653-71 750-987 682-95 691-93	1022 891-1011 878-999 653-42 716-972 721-857 743-956	842 725-80 705-88 653-42 671-87 676-86 657-57	932 797-850 707-864 653-71 750-987 676-86 694-700	1022 855-988 734 774-995 716-972 721-857 745-83	842 734 707-850 651-58 642-35 655-59 651-53	142 734 707-850 651-58 642-35 671-76 659-91	143 734 707-850 651-58 642-35 671-76 730-28	144-1 734 707-850 651-58 642-35 671-76 730-28	144-2 734 707-850 651-58 642-35 671-76 730-28	108 734 707-850 651-58 642-35 671-76 730-28	109 734 707-850 651-58 642-35 671-76 730-28	110 734 707-850 651-58 642-35 671-76 730-28	114 734 707-850 651-58 644-50 687-91	115 734 707-850 651-58 644-50 687-91	116-1 734 707-850 651-58 644-50 687-91	116-2 734 707-850 651-58 644-50 687-91
0.0 24.6 41.6 6.0 27.8 0.0	0.4 22.5 53.9 4.4 15.3 5.9	0.5 20.5 52.1 5.3 11.3 5.0	0.6 26.2 50.6 6.0 12.3 4.3	1.4 23.3 55.6 5.6 7.9 7.4	0.0 29.1 41.5 9.4 20.0 0.0	0.0 24.6 7.7 7.4 28.2 0.2	0.4 22.6 46.5 5.8 20.6 4.0	0.0 27.8 42.3 9.5 30.0 0.1	0.0 25.3 37.2 7.3 30.0 0.2	0.2 22.1 37.0 5.5 20.0 1.6	0.0 28.7 41.6 9.7 28.9 0.0	0.0 26.4 36.8 7.9 28.9 0.0	0.0 22.7 34.1 7.2 36.3 0.0	1.1 17.4 39.0 7.4 30.2 4.9	0.3 30.0 40.1 5.9 13.9 1.8	0.7 27.0 46.3 6.1 16.0 3.4	1.4 22.5 51.5 6.2 11.6 6.8	0.0 27.2 42.1 10.2 20.5 0.0	0.0 25.2 36.5 7.9 30.4 0.0	0.2 22.8 35.3 5.5 36.2 0.0	4.0 21.5 24.1 3.5 7.6 9.3	
55.4 100 92	25.2 81.9 248	19.2 82.8 140	22.8 78.5 191	20.7 63.3 194	29.0 100 22	55.2 77.4 252	30.4 85.5 18	29.8 99.7 43	37.9 99.2 43	40.5 95.6 256	29.3 100 17	36.4 100 45	42.9 100 154	43.2 86.1 272	21.5 90.2 123	25.8 84.1 215	25.6 68.0 --	30.3 100 14	37.9 100 38	41.3 99.8 238	24.0 43.8 281	

129	130	131	132-1	132-2	133-1	121	122	123	124-1	124-2	134	135	136-1	136-2	137-2
			10280-119C						10330-124C						10280-157B
1202 977-1035 892-1121 770-73 797-804 853-33	842 640-42 650-42 621-26 628-32 640-42	932 660 662-76 651-55 662 680	1022 729-27 700-16 684-87 700-698 729-27	1112 786-79 738-61 725 747-43 785-79	1202 860-38 842-1013 788-803 812-01 860-38	842 732-38 662-82 551-62 664-71 662-87	932 797-800 705-35 691-102 709-14 797-802	1022 647-69 748-776 736-47 763-66 799	1112 948-50 799-838 791-801 630-31 880-76	1202 1058-1179 966-1157 896-1150 923-1146 981-1146	842 925-27 637-53 676-98 635-40 644-51 725-27	932 786-90 676-98 673-80 684-89 707-12	1022 851-55 716-43 709-18 759-61 756-58	1112 923 763-94 759-61 779-77 817-1	1202 1006-02 833-91 817-24 842-38 885-78
3.2 12.7 14.1 4.1 64.7 1.2	0.1 34.8 27.4 14.6 23.4 0.0	0.5 31.0 21.6 11.6 35.5 0.0	0.5 26.7 16.6 7.9 48.0 0.1	1.2 20.8 11.6 5.2 60.7 0.3	3.6 13.1 9.4 3.9 69.0 0.3	0.6 27.3 47.0 5.6 19.5 0.2	1.2 25.9 39.7 4.7 28.3 0.0	2.2 23.3 33.3 4.6 35.8 0.6	3.2 19.1 27.5 5.4 42.5 2.3	7.6 16.9 36.6 7.9 25.4 5.6	0.0 30.3 27.9 39.5 7.3 22.9 0.0	0.0 27.9 33.8 28.1 6.1 32.2 0.0	0.2 24.3 28.1 4.4 42.8 0.2	0.3 19.6 23.7 3.2 53.0 0.2	1.9 13.9 17.8 2.5 63.4 0.5
72.8 94.0 229	47.0 99.4 15	56.3 98.6 14	67.0 97.6 16	77.1 94.0 23	77.1 94.0 171	25.3 97.6 20	34.0 95.9 29	43.2 92.6 27	53.4 89.0 40	46.5 70.8 234	29.8 100 16	37.9 100 22	47.2 99.2 31	56.3 98.1 58	67.9 96.5

Table 55 (Contd).

Table 56. DEHYDROGENATIONS OF METHYLCYCLOHEXANE OVER VARIOUS CATALYSTS

## Catalyst Stability Tests:

Feed: Pure MOH  
Catalyst Volume: 7 ml  
Pressure: 1 atm  
Block Temperature: 842°F  
Reaction Time at Each LHSV: 30 minutes

年	月	日	星期	姓名	性别	年龄	籍贯	职业	住址	备注
1949	1	1	星期一	王德胜	男	25	山东	工人	天津	
1949	1	2	星期二	李德胜	男	25	山东	工人	天津	
1949	1	3	星期三	张德胜	男	25	山东	工人	天津	
1949	1	4	星期四	赵德胜	男	25	山东	工人	天津	
1949	1	5	星期五	刘德胜	男	25	山东	工人	天津	
1949	1	6	星期六	孙德胜	男	25	山东	工人	天津	
1949	1	7	星期日	周德胜	男	25	山东	工人	天津	
1949	1	8	星期一	吴德胜	男	25	山东	工人	天津	
1949	1	9	星期二	郑德胜	男	25	山东	工人	天津	
1949	1	10	星期三	冯德胜	男	25	山东	工人	天津	
1949	1	11	星期四	陈德胜	男	25	山东	工人	天津	
1949	1	12	星期五	林德胜	男	25	山东	工人	天津	
1949	1	13	星期六	黄德胜	男	25	山东	工人	天津	
1949	1	14	星期日	周德胜	男	25	山东	工人	天津	
1949	1	15	星期一	吴德胜	男	25	山东	工人	天津	
1949	1	16	星期二	郑德胜	男	25	山东	工人	天津	
1949	1	17	星期三	冯德胜	男	25	山东	工人	天津	
1949	1	18	星期四	陈德胜	男	25	山东	工人	天津	
1949	1	19	星期五	林德胜	男	25	山东	工人	天津	
1949	1	20	星期六	黄德胜	男	25	山东	工人	天津	
1949	1	21	星期日	周德胜	男	25	山东	工人	天津	
1949	1	22	星期一	吴德胜	男	25	山东	工人	天津	
1949	1	23	星期二	郑德胜	男	25	山东	工人	天津	
1949	1	24	星期三	冯德胜	男	25	山东	工人	天津	
1949	1	25	星期四	陈德胜	男	25	山东	工人	天津	
1949	1	26	星期五	林德胜	男	25	山东	工人	天津	
1949	1	27	星期六	黄德胜	男	25	山东	工人	天津	
1949	1	28	星期日	周德胜	男	25	山东	工人	天津	
1949	1	29	星期一	吴德胜	男	25	山东	工人	天津	
1949	1	30	星期二	郑德胜	男	25	山东	工人	天津	
1949	1	31	星期三	冯德胜	男	25	山东	工人	天津	

姓名	性别	年龄	籍贯	职业	文化程度	健康状况	婚姻状况	子女情况	其他
张三	男	45	山东	工人	小学	良好	已婚	2子1女	
李四	女	35	河北	家庭主妇	小学	良好	已婚	1子1女	
王五	男	55	河南	农民	小学	良好	已婚	3子2女	
赵六	男	65	山西	退休	小学	良好	已婚	4子3女	
孙七	女	75	江苏	退休	小学	良好	已婚	5子4女	
周八	男	85	浙江	退休	小学	良好	已婚	6子5女	
吴九	女	95	安徽	退休	小学	良好	已婚	7子6女	
郑十	男	105	江西	退休	小学	良好	已婚	8子7女	
钱十一	女	115	福建	退休	小学	良好	已婚	9子8女	
陈十二	男	125	广东	退休	小学	良好	已婚	10子9女	
林十三	女	135	广西	退休	小学	良好	已婚	11子10女	
黄十四	男	145	四川	退休	小学	良好	已婚	12子11女	
宋十五	女	155	湖南	退休	小学	良好	已婚	13子12女	
李十六	男	165	湖北	退休	小学	良好	已婚	14子13女	
王十七	女	175	陕西	退休	小学	良好	已婚	15子14女	
张十八	男	185	甘肃	退休	小学	良好	已婚	16子15女	
赵十九	女	195	宁夏	退休	小学	良好	已婚	17子16女	
孙二十	男	205	青海	退休	小学	良好	已婚	18子17女	
周二十一	女	215	新疆	退休	小学	良好	已婚	19子18女	
吴二十二	男	225	内蒙古	退休	小学	良好	已婚	20子19女	
郑二十三	女	235	吉林	退休	小学	良好	已婚	21子20女	
钱二十四	男	245	辽宁	退休	小学	良好	已婚	22子21女	
陈二十五	女	255	黑龙江	退休	小学	良好	已婚	23子22女	
林二十六	男	265	河北	退休	小学	良好	已婚	24子23女	
黄二十七	女	275	山西	退休	小学	良好	已婚	25子24女	
宋二十八	男	285	山东	退休	小学	良好	已婚	26子25女	
李二十九	女	295	河南	退休	小学	良好	已婚	27子26女	
王三十	男	305	安徽	退休	小学	良好	已婚	28子27女	
张三十一	女	315	江西	退休	小学	良好	已婚	29子28女	
赵三十二	男	325	福建	退休	小学	良好	已婚	30子29女	
孙三十三	女	335	广东	退休	小学	良好	已婚	31子30女	
周三十四	男	345	广西	退休	小学	良好	已婚	32子31女	
吴三十五	女	355	四川	退休	小学	良好	已婚	33子32女	
郑三十六	男	365	湖南	退休	小学	良好	已婚	34子33女	
钱三十七	女	375	湖北	退休	小学	良好	已婚	35子34女	
陈三十八	男	385	陕西	退休	小学	良好	已婚	36子35女	
林三十九	女	395	甘肃	退休	小学	良好	已婚	37子36女	
黄四十	男	405	宁夏	退休	小学	良好	已婚	38子37女	
宋四十一	女	415	青海	退休	小学	良好	已婚	39子38女	
李四十二	男	425	新疆	退休	小学	良好	已婚	40子39女	
王四十三	女	435	内蒙古	退休	小学	良好	已婚	41子40女	

姓名	性别	年龄	籍贯	职业	文化程度	健康状况	婚姻状况	家庭成员	备注
王德胜	男	45	山东烟台	工人	小学	健康	已婚	妻：李氏，子：王小明	
张子安	男	38	河北保定	农民	初中	健康	已婚	妻：张王氏，子：张子明	
李德胜	男	52	河南开封	商人	小学	健康	已婚	妻：李氏，子：李德明	
赵子安	男	40	山西太原	工人	小学	健康	已婚	妻：赵王氏，子：赵子明	
孙德胜	男	48	江苏苏州	商人	小学	健康	已婚	妻：孙氏，子：孙德明	
周子安	男	35	浙江杭州	工人	小学	健康	已婚	妻：周王氏，子：周子明	
吴德胜	男	50	安徽合肥	商人	小学	健康	已婚	妻：吴氏，子：吴德明	
郑子安	男	42	江西九江	工人	小学	健康	已婚	妻：郑王氏，子：郑子明	
冯德胜	男	46	湖南长沙	商人	小学	健康	已婚	妻：冯氏，子：冯德明	
陈子安	男	39	四川成都	工人	小学	健康	已婚	妻：陈王氏，子：陈子明	
何德胜	男	51	广东广州	商人	小学	健康	已婚	妻：何氏，子：何德明	
周子安	男	41	福建福州	工人	小学	健康	已婚	妻：周王氏，子：周子明	
吴德胜	男	49	广西梧州	商人	小学	健康	已婚	妻：吴氏，子：吴德明	
郑子安	男	43	云南昆明	工人	小学	健康	已婚	妻：郑王氏，子：郑子明	
冯德胜	男	47	贵州贵阳	商人	小学	健康	已婚	妻：冯氏，子：冯德明	
陈子安	男	40	陕西西安	工人	小学	健康	已婚	妻：陈王氏，子：陈子明	
何德胜	男	53	甘肃兰州	商人	小学	健康	已婚	妻：何氏，子：何德明	
周子安	男	44	宁夏银川	工人	小学	健康	已婚	妻：周王氏，子：周子明	
吴德胜	男	50	青海西宁	商人	小学	健康	已婚	妻：吴氏，子：吴德明	
郑子安	男	45	新疆乌鲁木齐	工人	小学	健康	已婚	妻：郑王氏，子：郑子明	
冯德胜	男	48	内蒙古呼和浩特	商人					

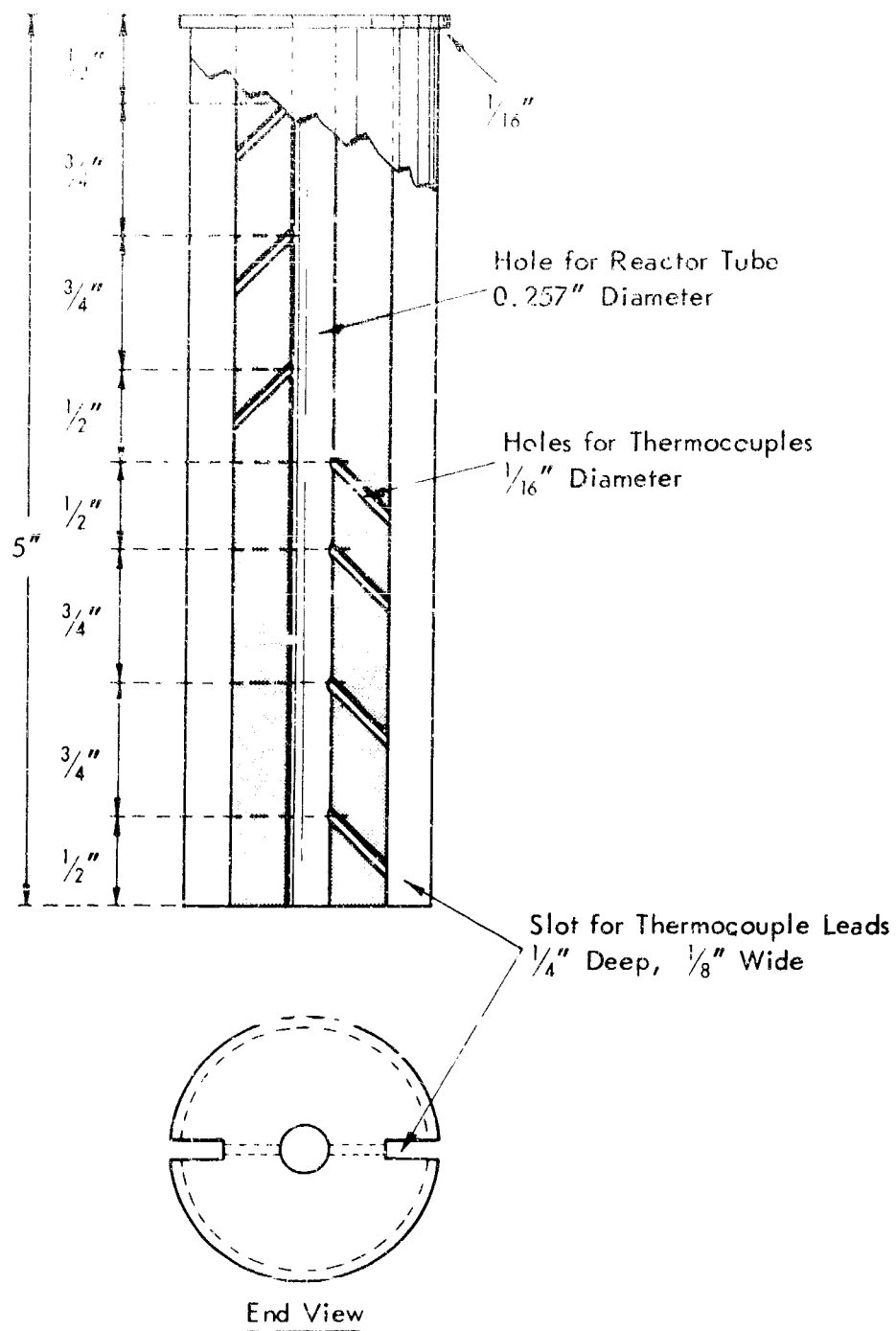


Figure 66. SECONDARY FURNACE LINER FOR PULSE REACTOR

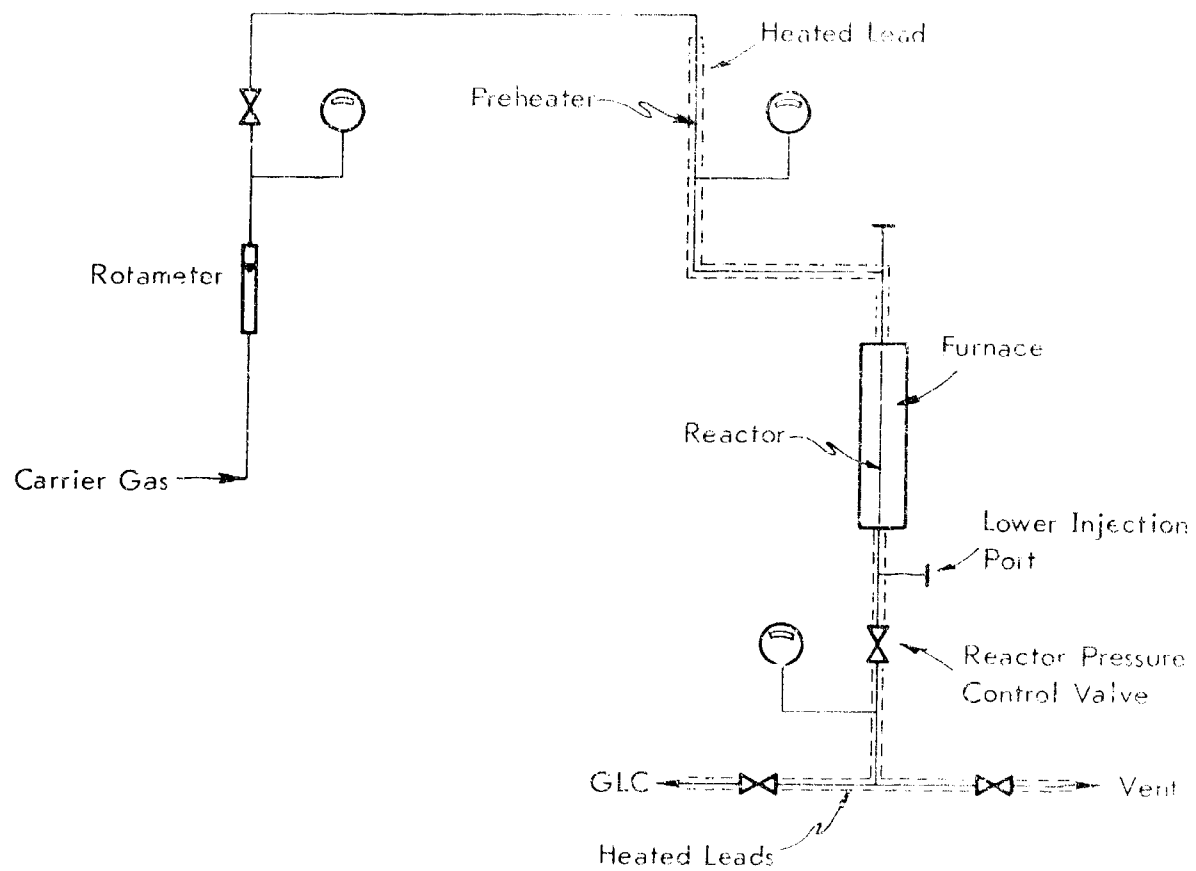


Figure 67. SCHEMATIC DIAGRAM OF PULSE REACTOR

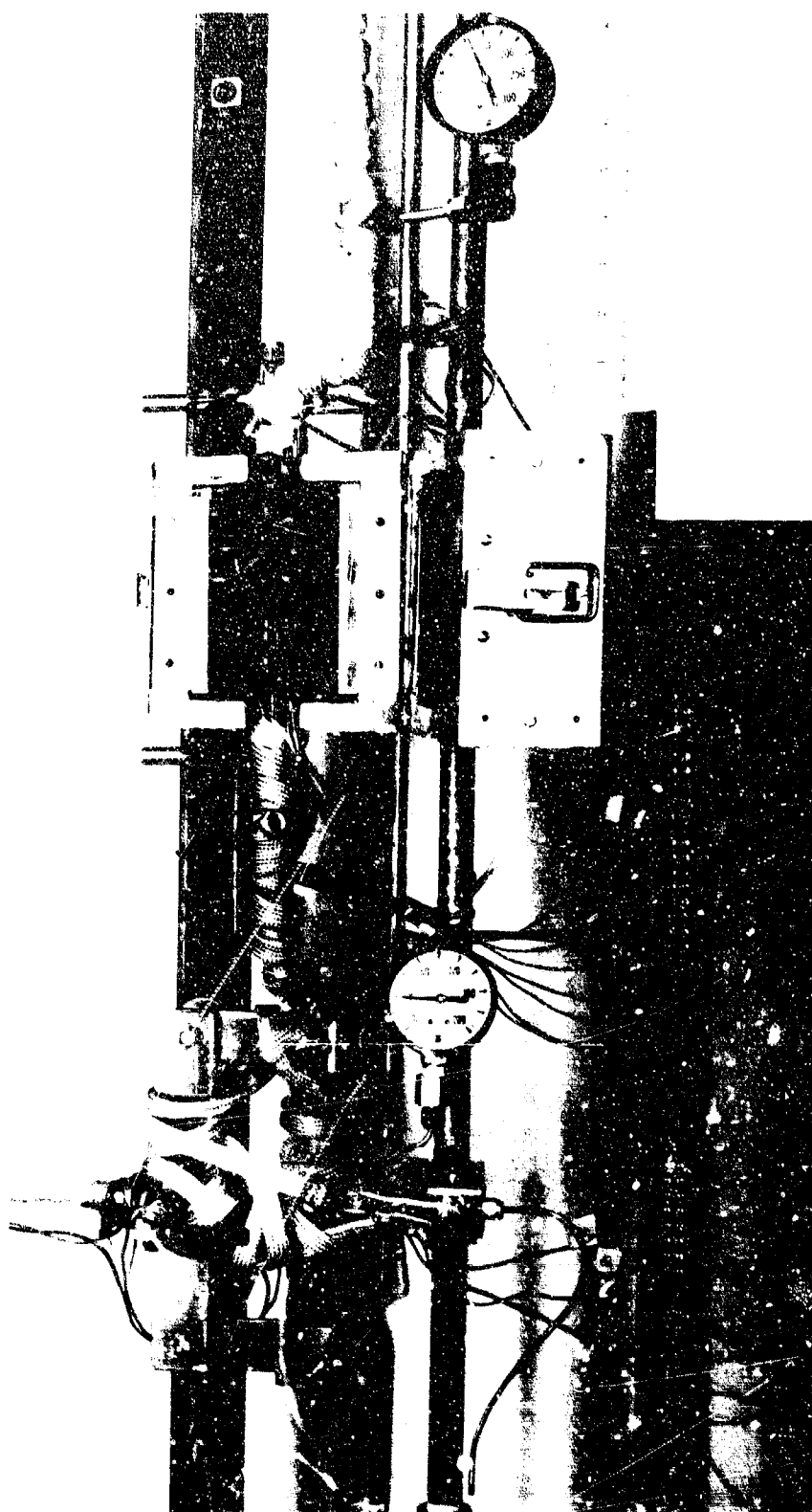


Figure 68. PULSE REACTOR SYSTEM

AFAPL-TR-70-71  
64286-5

### Calculation of Rate Constants (Bench-Scale Reactor)

First order rate constants were calculated based on the rate of disappearance of the starting material according to the following equation:

$$k = \frac{\text{LHSV}}{3600} \times \frac{\rho \times 22,412}{\text{MW} \times P} \times \frac{T}{273} \times 2.3 \log \left( \frac{1}{1-f} \right) \quad (35)$$

where:  $k$  = first-order rate constant in  $\text{sec}^{-1}$

LHSV = liquid hourly space velocity (i.e., volumes of feed/volume of catalyst bed per hour)

MW = molecular weight

$P$  = reactor pressure in atmospheres

$T$  = reaction temperature in  $^{\circ}\text{K}$  (reactor wall temperature)

$\rho$  = liquid density

$f$  = fraction component reacted

### Calculation of Reaction Products for the Thermal Reaction of Bicyclo (2,2,2)octane

Both BCO and TMB react thermally to form reaction products 1, 2, 3 and 4 (Tables 4 and 5). With BCO in TMB feed the amount of component A formed from TMB only is given by

$$X_A = \frac{C_T X_A^O}{C_T^O} \quad (36)$$

where  $X_A$  = amount component A formed with BCO-TMB feed

$C_T$  = fraction TMB reacted with BCO-TMB feed

$X_A^O$  = amount component A formed with pure TMB feed

$C_T^O$  = fraction TMB reacted with pure TMB feed

$C_T$  and  $C_T^O$  are obtained at the same temperature and space velocity.  $X_A^O$  and  $C_T^O$  are obtained from Table 4.

As an example, consider component 4 in Runs 102-5 (Table 4) and 105-2 (Table 5):

$$X_4^O = 18.7$$

$$C_T^O = 24.5$$

$$C_T = 11.1$$



$$X_4 = \frac{11.1(18.7)}{24.5} = 8.5$$

Since the total amount of component 4 found by GLC was only 8.0%, all of it was formed from TMB. Hence, none of component 4 was formed from BCO.

#### Micro Catalyst Test Reactor

The Micro Catalyst Test Reactor (MICTR) and the operational techniques used for screening candidate catalysts have been described in previous reports.<sup>1,2</sup> No further changes have been made. Figures 87 through 89 of reference 1 show the apparatus in detail, except for changes noted in reference 2. Catalysts have been tested with MCH at LHSV 100, at 662, 752, and 842°F, and at 10 atm pressure without added hydrogen. It has been found that more consistent results are obtained if a fresh loading of the reference catalyst 9874-139, or its replacement catalyst 10860-70, is tested each week as a base point for calibration, rather than using the same reference tube over and over again, since the activity gradually declines. Also, prepared catalysts have been rescreened to 10-20 mesh to remove fines after impregnation and drying of the supports, and this gives more reproducible results. Tables 57 and 58 give the MICTR test data in chronological order.

Tables 57 and 58 follow

Table 57. DEHYDROGENATION OF METHYLCYCLOHEXANE WITH VARIOUS CATALYSTS II. MICTR

Runs 1061-1191

Conditions: 100 LHSV, 10 atm pressure, no added H<sub>2</sub>, temperature variable. 0.9 ml catalysts diluted with 1.1 ml quartz chips (10-20 mesh) reduced in H<sub>2</sub> at 796°F. GLC samples normally taken at 3-, and 8-, and 13 minutes operation at each temperature.

Run No.	No.	Catalyst Description	Wt., g	% Conversion of MCH to Toluene, %	842°F
1061	987A-139	15 Pt/100 R-8 type Al <sub>2</sub> O <sub>3</sub> (ref.)	0.433	28, 24, 24	75, 71, 70
1062	6846-47	Bismuth, 75, 27% (unreported)	0.238	0.0	0.0
1063	619C-76	Monometallic, 47% Pt	0.305	0.0	0.0
1064	1003-41A	Bismuth, 95, 5% (unreported)	0.749	0.0	0.0
1065	6707-46	Triethylenic, 57, 29, 14% (unreported)	0.749	0.0	0.0
1066	6707-145	Quad metallic, 5, 27, 27, 41% (unreported)	0.755	0.0	0.0
1067	6707-139	15 Pt/100 R-8 type Al <sub>2</sub> O <sub>3</sub> (ref.)	0.433	28, 26, 26	76, 73, 71
1068	6709-11	Triethylenic, 11, 2, 87% (unreported)	1.022	0.0	5.2, 2
1069	6846-19A	Triethylenic, 25, 41% (unreported)	0.697	0.0	0.0
1070	6846-19	Triethylenic, 69, 31% (unreported)	0.320	0.0	0.0
1071	6846-26	Triethylenic, 54, 2, 44% (unreported)	0.700	0.0	0.0
1072	6187-168 D	Triethylenic, 58, 41, 1% (unreported)	0.633	0.0	0.0
1073	6849-49	Triethylenic, 46, 2, 52% (unreported)	0.681	0.0	0.0
1074	9073-10	Triethylenic, 54, 26, 20% (unreported)	0.760	0.0	0.0
1075	6749-18	Triethylenic, 9, 2, 89% (unreported)	0.879	0.0	0.0
1076	6749-13	Triethylenic, 7, 2, 91% (unreported)	0.773	0.0	0.0
1077	7003-17A	Bismuth, 2, 24% type 1 support	0.433	26, 26, 26	75, 73, 71
1078	987A-139	15 Pt/100 R-8 type Al <sub>2</sub> O <sub>3</sub> (ref.)	0.433	26, 26, 26	75, 73, 71
1079	6707-35	Quad metallic, 6, 41, 10 (unreported)	0.461	0.0	0.0
1080	6707-35	Quad metallic, 78, 20% (unreported)	0.498	0.0	0.0
1081	6846-30	Triethylenic, 46, 4, 49% (unreported)	0.544	0.0	0.0
1082	6749-32A	Triethylenic, 3, 4, 93% (unreported)	1.147	0.0	0.0
1083	10860-119A	24 Pt/40% type 1 support (1), 45% type 1 support (2), 20% type 22 binder	0.357	20, 23, 24	79, 76, 78
1084	10860-119B	24 Pt/40% type 1 support (1), 45% type 1 support (2), 20% type 22 binder	0.398	31, 26, 23	79, 77, 75
1085	10860-119C	24 Pt/45% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.555	26, 24, 24	85, 81, 80
1086	10860-119D	24 Pt/45% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.550	27, 25, 27	88, 85, 88
1087	10860-118	24 Pt/45% type 1 support (1), 45% type 1 support (2), 10% type 22 binder, coated tube No. 28	0.046	26, 23, 20	8, 7, 9
1088	10860-122	Alumina: 1/4" tube	0.433	27, 27, 26	78, 73, 71
1089	987A-139	15 Pt/100 R-8 type Al <sub>2</sub> O <sub>3</sub> (ref.)	0.350	28, 29, 29	81, 80, 78
1090	10860-121	24 Pt/100 R-8 type Al <sub>2</sub> O <sub>3</sub> (ref.)	0.367	28, 25, 24	73, 74, 72
1091	10860-125A	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.472	20, 21, 21	81, 79, 86
1092	10860-125B	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.468	24, 23, 20	76, 76, 76
1093	10860-125C	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.468	20, (28), 23	86, 84, 85
1094	10860-125D	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1095	10860-125E	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1096	10860-125F	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1097	10860-125G	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1098	10860-125H	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1099	10860-125I	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1100	10860-125J	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1101	10860-125K	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1102	10860-125L	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1103	10860-125M	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1104	10860-125N	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1105	10860-125O	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1106	10860-125P	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1107	10860-125Q	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1108	10860-125R	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1109	10860-125S	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1110	10860-125T	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1111	10860-125U	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1112	10860-125V	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1113	10860-125W	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1114	10860-125X	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1115	10860-125Y	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1116	10860-125Z	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1117	10860-126A	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1118	10860-126B	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1119	10860-126C	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1120	10860-126D	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1121	10860-126E	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1122	10860-126F	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1123	10860-126G	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1124	10860-126H	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1125	10860-126I	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1126	10860-126J	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1127	10860-126K	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1128	10860-126L	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1129	10860-126M	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1130	10860-126N	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1131	10860-126O	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1132	10860-126P	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1133	10860-126Q	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1134	10860-126R	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1135	10860-126S	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1136	10860-126T	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1137	10860-126U	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1138	10860-126V	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1139	10860-126W	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1140	10860-126X	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1141	10860-126Y	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1142	10860-126Z	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1143	10860-127A	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1144	10860-127B	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1145	10860-127C	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1146	10860-127D	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1147	10860-127E	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1148	10860-127F	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1149	10860-127G	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1150	10860-127H	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1151	10860-127I	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1152	10860-127J	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1153	10860-127K	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1154	10860-127L	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1155	10860-127M	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1156	10860-127N	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1157	10860-127O	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1158	10860-127P	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1159	10860-127Q	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1160	10860-127R	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1161	10860-127S	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1162	10860-127T	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1163	10860-127U	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1164	10860-127V	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1165	10860-127W	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1166	10860-127X	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1167	10860-127Y	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1168	10860-127Z	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1169	10860-128A	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
1170	10860-128B	24 Pt/40% type 1 support (1), 45% type 1 support (2), 10% type 22 binder	0.477	30, 25, 25	86, 84, 85
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Table 58. DEHYDROGENATION OF METHYLCYCLOHEXANE WITH VARIOUS CATALYSTS IN MICTR  
Runs 1192-1276

Conditions: 100 LHSV, 10 atm pressure, no added H<sub>2</sub>, temperature variable. 0.9 ml catalysts diluted with 1.1 ml quartz chips (10-20 mesh) reduced in H<sub>2</sub> at 796°F. GLC samples normally taken at 3-, and 8-, and 13 minutes operation at each temperature.

Run No.	Catalyst			% Conversion of MCH to Toluene, %		
	No. 10860-	Description	wt., g.	662	752	842°F
1192	70	1% Pt/UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	0.411	23, 27, 25	51, 48, 48	73, 72, 72
1193	163A	3.2% Pt/80% type 1 support <sup>a)</sup> 20% type 6 binder	0.346	--, 26, 25	50, 50, 50	82, 81, 80
1194	163B	3.2% Pt/40% type 1 support (1) <sup>a)</sup> 40% type 1 support (2) <sup>a)</sup> 20% type 6 binder	0.371	26, 23, 23	54, 50, 49	81, 77, 77
1195	163C	1.6% Pt/40% type 1 support (1) <sup>a)</sup> 40% type 1 support (2) 20% type 6 binder	0.403	23, 23, 23	55, 53, 49	77, 76, 75
1196	160A	1% Pt/80% type 16 support 20% type 6 binder	0.346	23, 21, 19	50, 47, 47	74, 74, 73
1197	160B	2% Pt/ " " "	0.399	25, 24, 23	50, 47, 47	74, 72, 72
1198	160C	4% Pt/ " " "	0.371	25, 25, 25	57, 54, 55	83, 82, 82
1199	161A	3.2% Pt/80% type 1 support <sup>a)</sup> 20% type 6 binder	0.594	23, 20, 21	52, 45, 45	75, 73, 73
1200	161B	1.6% Pt/40% type 1 support (1) 40% type 1 support (2) <sup>a)</sup> 20% type 6 binder	0.442	21, 23, 22	50, 46, 46	77, 75, 74
1201	70	1% Pt/UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	0.410	26, 21, 22	48, 46, 46	72, 71, 70
1202	157A	1% Pt/type 1 support <sup>i)</sup>	0.562	24, 22, 22	55, 50, 50	79, 78, 78
1203	157E	4% Pt/ " " " i)	0.596	26, 25, 25	60, 58, 58	85, 82, 83
1204	157B	1% Pt/type 1 support <sup>i)</sup>	0.545	25, 21, 23	53, 52, 52	81, 81, 79
1205	157F	4% Pt/ " " " i)	0.562	28, 26, 27	60, 59, 56	87, 87, 87
1206	158A	1% Pt/type 1 support	0.535	27, 25, 24	56, 53, 53	84, 83, 83
1207	158B	2% Pt/ " " "	0.542	29, 27, 24	54, 53, 51	81, 80, 82
1208	158C	4% Pt/ " " "	0.567	32, 24, 24	57, 53, 53	83, 83, 83
1209 <sup>b)</sup>	160B	2% Pt/80% type 16 support 20% type 6 binder	0.338	23, 23, 19	45, 43, 43	67, 65, 66
1210	157C	1% Pt/type 1 support <sup>i)</sup>	0.843	19, 18, 17	36, 35, 37	46, 48, 50
1211	157G	4% Pt/ " " " i)	0.828	17, 18, 18	36, 35, 35	46, 46, 47
1212	157D	1% Pt/type 1 support <sup>i)</sup>	0.527	25, 22, 22	48, 46, 46	74, 74, 74
1213	157H	4% Pt/ " " " i)	0.548	23, 23, 22	53, 50, 49	78, 77, 77
1214	70	1% Pt/UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	0.412	24, 20, 23	50, 45, 46	70, 69, 70
1215	164A	1% Pt/80% type 1 support 20% type 6 binder	0.430	22, 21, 22	50, 49, 48	76, 75, 76
1216	164B	2% Pt/ " " "	0.431	30, 28, 26	53, 54, 56	84, 83, 84
1217	164C	3% Pt/ " " "	0.458	30, 27, 27	59, 56, 57	85, 85, 85
1218	165 <sup>c)</sup>	4% Pt/type 1 support	0.549	25, 29, 29	48, 51, 51	80, 79, 80
1219	165A <sup>c)</sup>	1% Pt/ " " "	0.534	24, 27, 25	59, 59, 54	83, 81, 81
1220	166A	4% Pt/type 6 support	0.321	28, 22, 22	47, 43, 42	65, 72, 65
1221	166B	4% Pt/type 6 support	0.325	20, 22, 20	45, 43, 43	67, 67, 65

a) Platinised with 4% metal before formulation.

b) Repeat of run 1197.

c) Repeat preparations in quantity of 10860-158C and 158A, for bench scale tests.

d) Acetate neutralised Pt(NH<sub>3</sub>)<sub>4</sub>(OH)<sub>2</sub>.

e) Impregnate type 31.

f) Repeat of run 1219.

g) Repeat of run 1218.

h) Repeat of run 1198.

i) In-house support.

(Continued)

**Table 58. (Contd-1) DEHYDROGENATION OF METHYLCYCLOHEXANE  
WITH VARIOUS CATALYSTS IN MICR**

Run No.	Catalyst			% Conversion of MCH to Toluene, %		
	No. 10860-	Description	wt., g.	662	752	842°F
1222	167A	4% Pt/80% type 16 support 20% type 6 binder	0.461	24, 25, 23	50, 41, 41	61, 59, 60
1223	165A <sup>1)</sup>	1% Pt/type 1 Support	0.528	23, 28, 22	57, 51, 53	82, 81, 81
1224	167B	4% Pt/type 16 support	0.384	24, 25, 23	50, 41, 41	61, 59, 60
1225	167C	3.2% Pt/80% type 16 support and type 6 binder	0.412	25, 20, 21	47, 43, 43	71, 69, 70
1226	165E <sup>1)</sup>	4% Pt/Type 1 support	0.555	25, 25, 22	56, 55, 54	85, 84, 82
1227	70	1% Pt/UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	0.413	21, 19, 23	48, 45, 45	71, 69, 69
1228	160C <sup>1)</sup>	4% Pt/80% type 16 support 20% type 6 binder	0.363	25, 22, 23	51, 48, 48	74, 72, 71
1229	168	1.6% Pt/40% type 1 support 40% type 1 support <sup>a)</sup> 20% type 1 binder	0.484	23, 22, 22	52, 49, 47	77, 74, 71
1230	172	1.6% Pt/40% type 16 support 40% type 1 support <sup>a)</sup> 20% type 6 binder	0.444	24, 20, 20	49, 47, 46	72, 71, 71
1231	170B	4% Pt/type 1 support <sup>1)</sup>	0.597	25, 22, 22	54, 51, 51	82, 81, 79
1232	70	1% Pt/UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	0.416	20, 22, 22	48, 45, 46	71, 69, 68
1233	171A	4% Pt/type 1 support	0.603	27, 23, 24	51, 49, 51	77, 77, 76
1234	171B	" " " "	0.606	25, 25, 25	58, 56, 57	86, 85, 83
1235	173	1.6% Pt/41% type 16 support, 41% type 1 support <sup>a)</sup> 18% type 6 binder	0.498	27, 25, 24	55, 52, 50	79, 77, 77
1236	170A	4% Pt/type 1 support <sup>1)</sup>	0.559	23, 24, 23	59, 57, 57	88, 87, 86
1237	171C	4% Pt/type 1 support <sup>1)</sup>	0.541	30, 27, 27	55, 55, 54	82, 82, 82
1238	171D	1% Pt/type 1 support	0.711	21, 21, 21	50, 47, 49	72, 69, 72
1239	171E	4% Pt/type 1 support	0.747	21, 22, 22	52, 51, 52	82, 82, 82
1240	171F	2% Pt/type 1 support	0.762	22, 20, 20	52, 48, 48	77, 76, 76
1241	175	3.2% Pt/40% type 16 support <sup>a)</sup> 40% type 1 support <sup>a)</sup> 20% type 6 binder	0.492	27, 22, 22	49, 45, 44	74, 74, 72
1242	"	" " " "	0.505	31, 22, 21	55, 50, 50	80, 77, 77
1243	70	1% Pt/UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	0.420	25, 20, 20	48, 45, 46	71, 71, 70
1244	177A	3% Pt/40% type 1 support (1) 40% type 1 support (2) 20% type 6 binder	0.405	23, 23, 24	53, 53, 55	82, 82, 82
1245	177B	4% Pt/ " " "	0.401	24, 24, 26	54, 53, 53	84, 82, 82
1246	177C	3% Pt/40% type 16 support 40% type 1 support 20% type 6 binder	0.478	29, 25, 27	54, 51, 50	79, 77, 77
1247	177D	4% Pt/ " " "	0.476	26, 24, 26	54, 51, 51	84, 80, 80
1248	184A	4% Pt/40% type 1 support (1) 40% type 1 support (2) 20% type 6 binder	0.404	30, 26, 25	58, 54, 55	85, 86, 83
1250	184C	4% Pt/80% type 1 support 20% type 6 binder	0.478	30, 25, 25	63, 60, 58	88, 89, 91

(Continued)

**Table 58. (Contd-2) DEHYDROGENATION OF METHYLCYCLOHEXANE  
WITH VARIOUS CATALYSTS IN MICTR**

Run No.	Catalyst			% Conversion of MCH to Toluene <i>in</i>		
	No. 10860-	Description	wt., g.	662	752	842°F
1251	184D	4% Pt/80% type 1 support 20% type 6 binder	0.664	26, 25, 29	57, 54, 54	84, 84, 83
1252	184E	4% Pt/40% type 1 support (1) 40% type 1 support (2) 20% type 6 binder	0.480	25, 27, 30	56, 53, 52	86, 85, 84
1253	184B	4% Pt/40% type 16 support 40% type 1 support 20% type 6 binder	0.422	22, 22, 25	51, 49, 50	79, 79, 78
1254	70	1% Pt/UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	0.419	24, 20, 20	48, 44, 46	71, 69, 68
1255	70	" " " " " "	0.422	21, 20, 20	47, 44, 44	69, 68, 68
1256	188A	1% Pt/type 1 support (purified) <sup>j)</sup>	0.803	31, 27, 26	58, 52, 51	84, 81, 80
1257	188B	1% Pt, 1% metal AB/type 1 support	0.738	21, 23, 24	60, 55, 55	86, 84, 84
1258	188C	" " 2.3% " " " "	0.713	28, 23, 22	56, 51, 51	80, 79, 79
1259	188D	" " 4% " " " "	0.752	16, 16, 16	48, 44, 45	71, 68, 68
1260	189A	1% Pt, 1% metal AC/type 1 support	0.759	31, 28, 30	57, 51, 51	82, 81, 81
1261	189B	" " 2.3% " " " "	0.731	17, 18, 20	51, 48, 49	73, 68, 67
1262	189C	" " 4% " " " "	0.747	16, 15, 15	37, 37, 32	52, 48, 45
1264	70	1% Pt/UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	0.417	24, 22, 20	49, 46, 46	72, 70, 69
1263	190A	1% Pt, 1% metal AA/type 1 support	0.711	25, 22, 25	59, 54, 53	84, 81, 81
1265	190B	" " 2.3% " " " "	0.767	20, 21, 24	55, 51, 50	81, 81, 80
1266	190C	" " 4% " " " "	0.764	22, 21, 19	53, 47, 47	78, 77, 75
1267	191A	1% Pt, 1% metal AD/type 1 support	0.767	34, 29, 26	59, 54, 54	83, 82, 81
1268	191B	" " 2.3% " " " "	0.746	24, 20, 21	59, 52, 54	85, 85, 83
1269	191C	" " 4% " " " "	0.742	22, 21, 23	51, 54, 51	73, 73, 72
1270	192A	1% Pt, 1% metal AE/type 1 support	0.736	26, 23, 25	62, 58, 56	87, 87, 86
1271	192B	" " 2.3% " " " "	0.737	26, 25, 24	59, 53, 52	85, 83, 83
1272	192C	" " 4% " " " "	0.769	26, 23, 22	55, 50, 50	81, 79, 79
1273	195C	Formulation for 10860-184C, stored wet 53 days, before drying, calcining, and 4% Pt impreg.	0.440	22, 23, 23	56, 55, 56	84, 82, 80
1274	195A	Formulation for 10860-184A, stored wet 55 days, before drying, calcining, and 4% Pt impreg.	0.409	24, 23, 25	54, 52, 51	84, 82, 79
1275	195E	Formulation for 10860-184E, stored wet 48 days, before drying, calcining and 4% Pt impreg.	0.503	24, 23, 23	58, 54, 53	84, 84, 83
1276	195D	Formulation for 10860-184D, stored wet 54 days, before drying, calcining, and 4% Pt impreg.	0.975	22, 22, 23	58, 54, 54	86, 84, 83

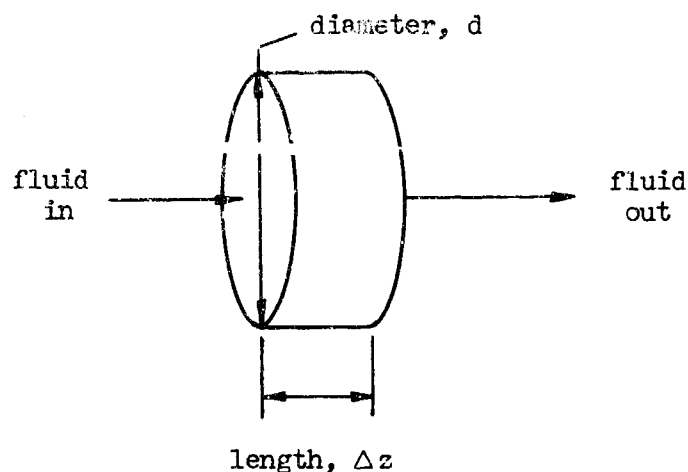
j) Control for series 10860-188, 189, 190, 191, and 192.

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## Derivations of Equations for the Catalytic Wall Reactor Model

### Fluid Flow Equations

The fluid flow in the reactor is described by a mass balance, a momentum balance, and an energy balance. The differential equations are formed by writing the balances over a differential cylindrical element:



#### Mass Balance:

Since the flow is assumed to be at steady state,  
rate of mass flow in = rate of mass flow out

$$\rho v A_c \Big|_z = \rho v A_c \Big|_{z + \Delta z} \quad (37)$$

where

$\rho$  = fluid density  
 $v$  = fluid velocity  
 $A_c$  = cross-sectional area =  $\frac{\pi d^2}{4}$   
 $d$  = diameter of flowing fluid

Since  $A_c$  is a constant, then  $\rho v$  is also a constant, which is the mass flux:

$$\boxed{G = \rho v} \quad (38)$$



Momentum Balance:

rate of momentum in - rate of momentum out + sum of forces on system = 0

$$\text{rate in} = (\rho v A_c) v|_z$$

$$\text{rate out} = (\rho v A_c) v|_{z + \Delta z}$$

$$\text{pressure force} = P A_c|_z - P A_c|_{z + \Delta z}$$

$$\text{drag force} = -(\frac{1}{2} \bar{\rho} \bar{v}^2) A_s f$$

where

$P$  = fluid pressure

$\bar{\rho}$  = average value of  $\rho$

$\bar{v}$  = average value of  $v$

$A_s$  = surface area of differential element

$f$  = Fanning friction factor (drag force equation defines  $f$ )

$$G A_c (v|_{z + \Delta z} - v|_z) + A_c (P|_z - P|_{z + \Delta z}) + \frac{1}{2} \bar{\rho} \bar{v}^2 A_s f = 0 \quad (39)$$

Dividing by the volume of the differential element and taking the limit as  $\Delta z$  approaches zero gives the desired differential equation:

$$\frac{G \left( \frac{\pi d^2}{4} \right) (v|_{z + \Delta z} - v|_z) - \frac{\pi d^2}{4} (P|_z - P|_{z + \Delta z}) + \frac{1}{2} \bar{\rho} \bar{v}^2 \pi d \Delta z f}{\frac{\pi d^2}{4} \Delta z} = 0 \quad (40)$$

$$\lim_{\Delta z \rightarrow 0} G \frac{v|_{z + \Delta z} - v|_z}{\Delta z} + \frac{P|_z - P|_{z + \Delta z}}{\Delta z} + \frac{2 \bar{\rho} \bar{v}^2 f}{d} = 0 \quad (41)$$

$$\boxed{G \frac{dv}{dz} + \frac{dP}{dz} + \frac{2 \bar{\rho} \bar{v}^2 f}{d} = 0} \quad (42)$$

Note that the average values of  $\rho$  and  $v$  become the same as the point values as  $\Delta z$  approaches zero. The only forces considered here are pressure and drag or frictional force. Other forces such as gravitational, nuclear, radiative, and electromagnetic forces have been neglected. The gravitational force might be important in a vertical reactor at very low flow rates.

Energy Balance:

rate of kinetic energy in + rate of internal energy in - rate of kinetic energy out - rate of internal energy out + net rate of heat added - net rate of work done by system = 0

$$\text{rate of kinetic energy in} = \frac{1}{2} G A_c v^2 \Big|_z$$

$$\text{rate of internal energy in} = G A_c U \Big|_z$$

$$\text{rate of kinetic energy out} = \frac{1}{2} G A_c v^2 \Big|_{z + \Delta z}$$

$$\text{rate of internal energy out} = G A_c U \Big|_{z + \Delta z}$$

$$\text{rate of heat added} = -q' A_s$$

$$\text{rate of work done by system} = P A_c v \Big|_{z + \Delta z} - P A_c v \Big|_z$$

where  $U$  = specific internal energy  
 $q'_1$  = heat flux from fluid to wall

Substituting  $H$  for  $U + P/\rho$ , and dividing by the volume element  $\pi d^2 \Delta z / 4$  gives

$$\frac{\frac{1}{2} G (v^2 \Big|_{z + \Delta z} - v^2 \Big|_z)}{\Delta z} + \frac{G (H \Big|_{z + \Delta z} - H \Big|_z)}{\Delta z} + \frac{4 q'_1}{d} = 0 \quad (45)$$

Taking the limit as  $\Delta z$  approaches zero and dividing by  $G$  gives

$$v \frac{dv}{dz} + \frac{dH}{dz} + \frac{4 q'_1}{G d} = 0 \quad (44)$$

We can obtain  $H$  in terms of  $P$ ,  $T$ , and  $\rho$ . For an ideal gas,  $H$  is a function of  $T$  only:

$$\frac{dH}{dz} = \left( \frac{\partial H}{\partial T} \right)_P \frac{dT}{dz} = c_P \frac{dT}{dz} \quad (45)$$

The pressure effect can be added to give:

$$\frac{dH}{dz} = \left( \frac{\partial H}{\partial T} \right)_P \frac{dT}{dz} + \left( \frac{\partial H}{\partial P} \right)_T \frac{dP}{dz} \quad (46)$$

The value of  $\left(\frac{\partial H}{\partial T}\right)_P$  is known as the heat capacity at constant pressure,  $c_p$ , and may be calculated. From the well known equation

$$dH = TdS + VdP \quad (47)$$

we can obtain

$$\left(\frac{\partial H}{\partial P}\right)_T = T \left(\frac{\partial S}{\partial P}\right)_T + V \quad (48)$$

Substituting in one of Maxwell's Relations, gives

$$\left(\frac{\partial H}{\partial P}\right)_T = -T \left(\frac{\partial V}{\partial T}\right)_P + V \quad (49)$$

or written in terms of  $\rho$  instead of  $V$ ,

$$\left(\frac{\partial H}{\partial P}\right)_T = -T \left(\frac{\partial \left(\frac{1}{\rho}\right)}{\partial T}\right)_P + \frac{1}{\rho} \quad (50)$$

$$= \frac{T}{\rho^2} \left(\frac{\partial \rho}{\partial T}\right)_P + \frac{1}{\rho} \quad (51)$$

So, the final equation is

$$\boxed{v \frac{dv}{dz} + c_p \frac{dT}{dz} + \left[ \frac{1}{\rho} + \frac{T}{\rho^2} \left(\frac{\partial \rho}{\partial T}\right)_P \right] \frac{dP}{dz} + \frac{4\alpha_1'}{3d} = 0} \quad (52)$$

We can rewrite the momentum balance by dividing by  $\rho$  to give

$$v \frac{dv}{dz} + \frac{1}{\rho} \frac{dP}{dz} + \frac{2v^2 f}{d} = 0 \quad (53)$$

Subtracting this from Equation (52) gives

$$c_p \frac{dT}{dz} + \frac{T}{\rho^2} \left(\frac{\partial \rho}{\partial T}\right)_P \frac{dP}{dz} + \frac{4\alpha_1'}{3d} - \frac{2v^2 f}{d} = 0 \quad (54)$$

Substituting  $G/\rho$  for  $v$  gives the final two equations for the fluid flow:

$$\frac{d}{dz} \left( \frac{\partial \rho}{\partial F} \frac{dP}{dz} \right) + \frac{d}{dz} \left( \frac{\partial \rho}{\partial T} \frac{dT}{dz} \right) - \frac{\rho d}{G^2} \frac{dP}{dz} - 2f = 0 \quad (55)$$

$$\frac{\rho^2 c_p d}{G^2} \frac{dT}{dz} + \frac{T d}{G^2} \frac{\partial \rho}{\partial T} \frac{dP}{dz} + \frac{4 \rho^2 g_1'}{G^3} - 2f = 0 \quad (56)$$

### Chemical Conversion

The model is presently based on a single chemical reaction. Again, the shell balance method is used to obtain a differential equation.

rate reactant in = rate reactant out + rate reacted

$$GA_c n|_z = GA_c n|_{z+\Delta z} + r v_c \Delta z \quad (57)$$

where

$n$  = number of moles of reactant per unit weight feed

$r$  = rate of reaction per unit volume of catalyst

$v_c$  = volume of catalyst per unit length reactor

giving

$$-\frac{dn}{dz} = \frac{4rv_c}{G\pi d^2} \quad (58)$$

The volume of catalyst per unit length for a cylindrical layer on the inside of a tube is

$$v_c = \frac{\pi}{4} (D^2 - d^2) \quad (59)$$

where

$D$  = inside diameter of tube

$d$  = inside diameter of catalyst

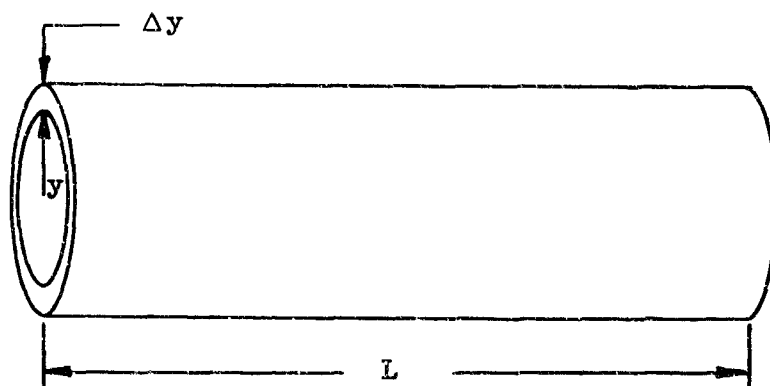
Rewriting Equation (58) in terms of  $x$ , number of moles converted per unit weight feed, gives

$$\boxed{\frac{dx}{dz} = \frac{r (D^2 - d^2)}{Gd^2}} \quad (60)$$

### Wall Temperature Profiles

The temperature profiles in the catalyst layer and in the tube wall may be calculated knowing the heat fluxes and assuming that the heat generation is uniform across the section. This is probably a good assumption for the tube wall, since the heat generated depends on the electrical resistance, which depends on the temperature. The changes in temperature across the tube wall are generally small enough to neglect for this purpose. The assumption of uniform heat absorption in the catalyst layer is less acceptable, since the amount of heat is related directly to the rate of reaction, which is an exponential function of temperature. The temperature drops across the catalyst layer are also larger than those across the tube wall.

The temperature in a cylindrical layer may be found by making a shell balance over an cylindrical element of inside radius  $y$ , thickness  $\Delta y$ , and length  $L$ :



The heat balance consists of

rate of heat in + rate of heat generated = rate of heat out

rate of heat in =  $2\pi y L q|_y$

rate of heat out =  $2\pi y L q|_{y + \Delta y}$

rate of heat generated =  $S(2\pi y L \Delta y)$

where

$y$  = radius of element

$\Delta y$  = thickness of element

$L$  = length of element

$q$  = heat flux in +  $y$  direction

$S$  = rate of heat generated per unit volume

$$2\pi L [(y + \Delta y) q|_{y + \Delta y} - y q|_y] = 2\pi y L \Delta y S \quad (61)$$

Divide by  $2\pi L \Delta y$  and take the limit as  $\Delta y$  approaches zero,

$$\lim_{\Delta y \rightarrow 0} \frac{[(y + \Delta y)q]_{y+\Delta y} - yq|_y}{\Delta y} = yS \quad (62)$$

$$\frac{d(yq)}{dy} = yS \quad (63)$$

Integration gives

$$yq = \frac{Sy^2}{2} + c_1 \quad (64)$$

$$\text{or } q = \frac{Sy}{2} + \frac{c_1}{y} \quad (65)$$

where  $c_1$  is a constant of integration. This equation applies to both layers. The boundary conditions are slightly different. In both cases, let  $y_0$  and  $y_i$  be the outer and inner radii of the layers, catalyst and tube. Then at the outer surface, the boundary condition is a heat flux at that surface:

$$q = q_0 \quad \text{at} \quad y = y_0$$

Then from Equation (65)

$$q_0 = \frac{Sy_0}{2} + \frac{c_1}{y_0} \quad (66)$$

$$\text{and } c_1 = q_0 y_0 - \frac{Sy_0^2}{2} \quad (67)$$

$$\text{So } q = \frac{Sy}{2} + \frac{1}{y} \left( q_0 y_0 - \frac{Sy_0^2}{2} \right) \quad (68)$$

Introducing the Fourier equation for heat conduction

$$q = -k \frac{dT}{dy} \quad (69)$$

$$\text{gives } \frac{dT}{dy} = -\frac{Sy}{2k} - \frac{1}{ky} \left( q_0 y_0 - \frac{Sy_0^2}{2} \right) \quad (70)$$

Integration gives

$$T = -\frac{Sy^2}{4k} - \frac{1}{k} \ln y \left( q_0 y_0 - \frac{Sy_0^2}{2} \right) + c_2 \quad (71)$$

where

$T$  = temperature in layer  
 $k$  = thermal conductivity  
 $c_2$  = constant of integration

The boundary condition for each layer is that at the inner surface,  $y_i$ ; the temperature is  $T_i$ .

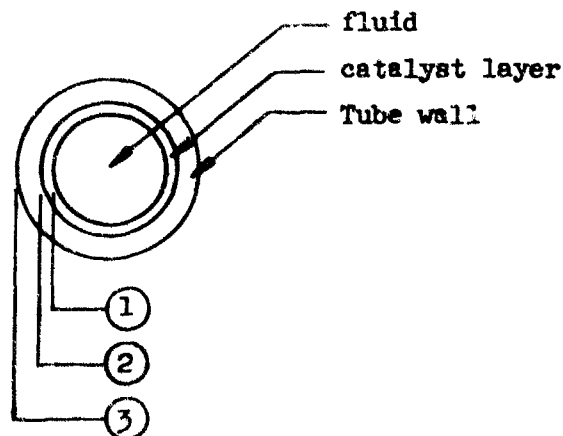
$$\text{So } T_i = -\frac{Sy_i^2}{4k} - \frac{1}{k} \ln y_i \left( q_0 y_0 - \frac{Sy_0^2}{2} \right) + c_2 \quad (72)$$

$$c_2 = T_i + \frac{Sy_i^2}{4k} + \frac{1}{k} \ln y_i \left( q_0 y_0 - \frac{Sy_0^2}{2} \right) \quad (73)$$

$$\begin{aligned} \text{giving } T = & -\frac{Sy^2}{4k} - \frac{(q_0 y_0 - \frac{Sy_0^2}{2})}{k} \ln y + T_i + \frac{Sy_i^2}{4k} \\ & + \frac{1}{k} \ln y_i (q_0 y_0 - \frac{Sy_0^2}{2}) \end{aligned} \quad (74)$$

for each layer.

Equation (74) may now be applied to each layer. The subscripts refer to the surfaces shown:



The heat generated in the tube wall is written as  $q_G'$ , the heat flux of generated heat, based on inner tube surface area:

$$S = q_G' \frac{\text{Btu}}{\text{hr ft}^2(\text{inner area})} \times \frac{\text{ft}^2(\text{inner area})}{\text{ft}^3(\text{wall})}$$

$$= q_G' \frac{2\pi y_2 L}{\pi(y_3^2 - y_2^2) L} \quad (75)$$

$$S = \frac{2y_2}{y_3^2 - y_2^2} q_G' \quad (76)$$

The heat flux at the outer surface (3) is  $q_3$  and is corrected to be based on the inner surface:

$$q_3 = \frac{y_2}{y_3} q_3' \quad (77)$$

Then Equation (74) after some rearrangement becomes

$$T - T_2 = \frac{1}{k} \left[ q_G' \left( \frac{\frac{y_2}{2y_3} (y_2^2 - y^2) + y_2 \ln \left( \frac{y}{y_2} \right)}{1 - \left( \frac{y_2}{y_3} \right)^2} \right) - q_3' y_2 \ln \left( \frac{y}{y_2} \right) \right] \quad (78)$$

To get the temperature drop,  $y_3$  and  $T_3$  are substituted for  $y$  and  $T$ , and rearranged to give

$$T_3 - T_2 = - \frac{q_G' y_2}{2k} \left[ \frac{1 - \left( \frac{y_2}{y_3} \right)^2 + 2 \ln \left( \frac{y_2}{y_3} \right)}{1 - \left( \frac{y_2}{y_3} \right)^2} \right] + \frac{q_3' y_2}{k} \ln \left( \frac{y_2}{y_3} \right) \quad (79)$$

for the tube wall. By a similar procedure, the temperature in the catalyst layer is shown to be

$$T - T_1 = + \frac{q_R' y_2}{2k} \left[ \frac{1 - \left( \frac{y}{y_1} \right)^2 + 2 \ln \left( \frac{y}{y_1} \right)}{1 - \left( \frac{y_2}{y_1} \right)^2} \right] - \frac{q_1' y_2}{k} \ln \left( \frac{y}{y_1} \right) \quad (80)$$

and the temperature difference across the catalyst layer is

$$T_2 - T_1 = \frac{q_R' y_2}{2k} \left[ \frac{1 - \left( \frac{y_2}{y_1} \right)^2 + 2 \ln \left( \frac{y_2}{y_1} \right)}{1 - \left( \frac{y_2}{y_1} \right)^2} \right] - \frac{q_1' y_2}{k} \ln \left( \frac{y_2}{y_1} \right) \quad (81)$$



### Description of the Beta-Ray Backscatter Apparatus for Rating Deposits

The instrument consists of the following functional blocks: The vacuum system, the scanning mechanism, the beta-ray source, and the detection system.

The vacuum system consists of a chamber made from a three-foot length of 6-inch sch 40 steel pipe with end plates sealed by O-rings. One end provides access for loading the coker tubes and the other holds the driving mechanism. A Welch 1402B fore pump is connected by rubber tubing to a pipe nipple welded to the vacuum chamber.

The rest of the vacuum system consists of a thermocouple gauge and a bleed valve.

The scanning mechanism serves to translate and rotate the coker tube past the source and detector. It consists of a track fastened to the inside of the vacuum chamber on which a carriage rides. The carriage holds the coker rod and is driven by a shaft and lead screw arrangement attached to the end plate of the vacuum system. The drive shaft in the test chamber connects through a vacuum seal to a variable speed reversible motor.

The beta-ray source is contained in a block which is mounted to the face of the detector. The source material was obtained from the target of a Texas Nuclear neutron generator. The block consists of layers of brass with two milled slots to direct the beta-ray beam to the coker tube, which is located at the intersection of the beams. Between the two slots for the beta-rays is a third slot which is aligned with the opening in the detector. This allows the backscattered radiation to enter the detector and be counted.

The detection system counts the radiation that is backscattered from the coker tube and presents the count rate on a strip chart recorder. The detector is a flow proportional counter with an ultra thin window operating on P-10 gas (90% Argon, 10% methane). The window is made of a thin film of cellulose nitrate. It is supported by a Buckbee Meers Co. nickel screen of 70% transmission. The detector is attached to a flange that provides a vacuum seal and can be moved while under vacuum to position the detector with the spout to the coker tube to focus the radiation.

The rest of the detection system is outside the vacuum chamber and consists of a high voltage power supply for the proportional counter, a low noise preamplifier, a linear amplifier, a count rate meter, and a strip chart recorder with an offset zero provision.

Table 59. IGNITION DELAY TIMES FOR DECALIN-OXYGEN-ARGON MIXTURES

Press, (atm)	Temp, (°K)	Delay, (μ sec)	Press, (atm)	Temp, (°K)	Delay, (μ sec)	Press, (atm)	Temp, (°K)	Delay, (μ sec)
90% Argon, ER = 0.2								
0.60	1440	110	1.08	1170	3380			
0.61	1390	200	1.20	1340	180			
0.67	1340	430	1.06	1190	3240			
0.61	1230	2680	1.28	1380	150			
0.63	1270	1230	1.44	1500	60			
0.64	1290	210	1.07	1190	2980			
0.60	1220	3790						
99% Argon, ER = 0.5								
0.55	1480	3040	0.91	1380	3450	1.56	1430	2010
0.53	1520	1570	0.94	1470	1740	1.61	1430	2360
0.54	1490	2220	0.91	1480	1830	1.50	1450	1400
0.53	1530	1040	0.92	1520	740	1.47	1460	1330
0.51	1640	300	0.91	1510	1160			
0.49	1600	490	0.90	1560	660			
			0.89	1550	560			
			0.88	1530	770			
			0.90	1560	530			
			0.92	1600	540			
			0.91	1600	270			

Table 60. IGNITION DELAY TIMES FOR  
TETRALIN-OXYGEN-ARGON MIXTURES

Press, (atm)	Temp, (°K)	Delay, (μ sec)	Press, (atm)	Temp, (°K)	Delay, (μ sec)
99% Argon, ER = 0.1					
0.61	1510	650	1.00	1440	1310
0.59	1540	540	1.02	1420	1480
0.61	1650	210	1.05	1420	2570
0.60	1700	350	0.97	1480	330
0.57	1760	170	0.98	1530	120
			0.95	1470	1070
80% Argon, ER = 0.1					
0.62	1210	3180	1.06	1140	3570
0.78	1470	40	1.08	1180	770
0.61	1210	880	1.04	1160	1970
0.62	1210	3110	1.02	1160	2730
0.62	1210	3130	1.06	1180	460
0.62	1210	2140	1.08	1190	370
0.61	1200	3390			
90% Argon, ER = 0.11					
0.62	1320	3470	1.04	1280	1050
0.62	1300	3520	1.03	1300	650
0.59	1320	1060	1.07	1370	290
0.63	1370	630	1.07	1280	1040
0.61	1320	1530	1.11	1280	1810
0.61	1390	250	1.14	1270	2960
0.68	1430	810	1.11	1210	3890
0.60	1300	2540			
99% Argon, ER = 0.5					
0.66	1520	3350	1.04	1490	2610
0.63	1540	1120	1.06	1530	930
0.65	1540	3070	1.05	1530	850
0.63	1520	1620	1.05	1520	790
0.65	1570	950	1.02	1560	410
0.64	1560	900	1.04	1680	260
0.63	1540	1150	1.05	1690	260
0.61	1570	820	1.04	1680	140
0.59	1630	460	1.04	1670	80
0.60	1720	240	1.10	1750	150
0.59	1700	220	1.05	1690	240
0.60	1720	120	1.13	1700	110
0.61	1740	210	1.05	1600	310
0.60	1710	180			
0.63	1530	3020			

(Contd)

Table 60. (Contd). IGNITION DELAY TIMES FOR  
TETRALIN-OXYGEN-ARGON MIXTURES

Press, (atm)	Temp, (°K)	Delay, (μ sec)	Press, (atm)	Temp, (°K)	Delay, (μ sec)
0.64	1560	890			
0.63	1550	1380			
0.62	1520	1170			
0.63	1530	1520			
90% Argon, ER = 0.57					
0.66	1390	60	1.14	1250	3580
0.62	1270	3310	1.14	1260	2700
0.61	1280	3470	1.13	1260	3650
0.60	1270	2660	1.09	1230	2590
0.59	1270	2070	1.08	1250	2620
			1.06	1240	2180
95% Argon, ER = 1.0					
0.58	1420	2880	1.08	1430	2280
0.62	1470	3040	1.03	1360	3130
0.61	1410	3340	1.03	1410	1640
0.67	1650	110	1.09	1450	190
0.62	1470	2630	1.09	1440	2360
0.63	1470	2900	1.04	1390	3130
0.63	1480	2480			
99% Argon, ER = 1.0					
0.59	1530	1650	1.01	1580	620
0.59	1560	760	0.97	1600	530
0.61	1640	450	1.01	1740	90
0.62	1730	250	1.03	1630	370
0.61	1750	230	0.99	1520	690
0.59	1630	370	1.02	1540	1520
0.62	1650	560	0.95	1510	1110
			1.03	1530	2890
90% Argon, ER = 1.21					
0.64	1280	2970	1.14	1230	3050
			1.14	1230	3290
			1.12	1230	2730
			1.12	1250	3450
			1.10	1250	3000

Table 61. IGNITION DELAY TIMES FOR  
NAPHTHALENE-OXYGEN-ARGON MIXTURES

Press, (atm)	Temp, (°K)	Delay, (μ sec)	Press, (atm)	Temp, (°K)	Delay, (μ sec)
90% Argon, ER = 0.1					
0.63	1430	400	1.04	1400	400
0.60	1470	100	1.05	1430	240
0.60	1370	730	1.04	1370	530
0.63	1360	890	1.08	1350	690
0.62	1330	3020	1.06	1270	2650
0.60	1300	3550	1.09	1280	2910
0.61	1310	3790			

Table 62. PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/ETHANE MIXTURES

		<u>MCH</u>	<u>Ethane</u>
	Weight %	90	10
	Mole %	73.4	26.6
Average Molecular Weight		80.1	
Melting Point		-215°F	
Heat of Formation (Liquid at 25°C)		-885 Btu/lb	
Net Heat of Combustion (25°C)		18,820 Btu/lb	
<u>Pseudocritical Properties</u>			
Pressure		538 psia	
Temperature		442°F	
Compressibility Factor		0.260	
Acentric Factor		0.207	

Temp., °F	Vapor Pressure, psia	Liquid Specific Gravity	Liquid Viscosity, cp	<u>Enthalpy, Btu/lb</u>	
				<u>0 psia</u>	<u>1000 psia</u>
-200	0.24	0.856	12.9	123	-62
-100	8.4	0.803	2.02	139	-36
0	59.	0.746	0.723	164	0
100				197	45
200				237	97
300				283	156
400				334	222
500				391	296
600				453	384
700				520	467
800				591	555
900				667	640
1000				746	725

Table 62 (Contd). PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/ETHANE MIXTURES

		<u>MCH</u>	<u>Ethane</u>
	Weight %	80	20
	Mole %	55.1	44.9
Average Molecular Weight		57.6	
Melting Point		-229°F	
Heat of Formation (Liquid at 25°C)		-936 Btu/lb	
Net Heat of Combustion (25°C)		18,980 Btu/lb	
<u>Pseudocritical Properties</u>			
Pressure		567 psia	
Temperature		354°F	
Compressibility Factor		0.266	
Acentric Factor		0.182	

Temp., °F	Vapor Pressure, psia	Liquid Specific Gravity	Liquid Viscosity, cp	<u>Enthalpy, Btu/lb</u>	
				<u>0 psia</u>	<u>1000 psia</u>
-200	0.40	0.816	5.04	112	-69
-100	14.2	0.759	1.07	139	-39
0	99.	0.696	0.470	165	0
100				200	48
200				240	102
300				265	164
400				339	231
500				397	306
600				459	393
700				527	477
800				598	565
900				675	649
1000				754	735

Table 63. PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/PROPANE MIXTURES

	<u>MCH</u>	<u>Propane</u>
Weight %	90	10
Mole %	80.2	19.8
Average Molecular Weight		87.5
Melting Point		-210°F
Heat of Formation (Liquid at 25°C)		-867 Btu/lb
Net Heat of Combustion (25°C)		18,770 Btu/lb
<u>Pseudocritical Properties</u>		
Pressure		520 psia
Temperature		498°F
Compressibility Factor		0.256
Acentric Factor		0.226

Temp., °F	Vapor Pressure, psia	Liquid Specific Gravity	Liquid Viscosity, cP	<u>Enthalpy, Btu/lb</u>	
				0 psia	1000 psia
-200		0.871	22.0	124	-60
-100	0.57	0.821	2.91	139	-35
0	7.5	0.769	0.898	164	0
100	38.1	0.713	0.430	197	44
200	124.	0.620	0.257	237	96
300				282	155
400				334	220
500				390	294
600				452	382
700				519	465
800				59	554
900				669	638
1000				744	723
1100				827	808
1200				913	896



Table 63 (Contd-1). PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/PROPANE MIXTURES

	<u>MCH</u>	<u>Propane</u>
Weight %	80	20
Mole %	64.2	35.8
Average Molecular Weight		78.8
Melting Point		-222°F
Heat of Formation (Liquid at 25°C)		-902 Btu/lb
Net Heat of Combustion (25°C)		18,880 Btu/lb
<u>Pseudocritical Properties</u>		
Pressure		536 psia
Temperature		440°F
Compressibility Factor		0.260
Acentric Factor		0.211

Temp., °F	Vapor Pressure, psia	Liquid Specific Gravity	Liquid Viscosity, cp	<u>Enthalpy, Btu/lb</u>	
				<u>0 psia</u>	<u>1000 psia</u>
-200		0.843	11.3	123	-64
-100	1.04	0.792	1.85	140	-37
0	13.5	0.737	0.646	166	0
100	67.6	0.675	0.340	199	46
200	215.	0.552	0.217	240	100
300				286	160
400				338	227
500				395	302
600				457	389
700				524	473
800				596	561
900				671	646
1000				751	731
1100				854	817
1200				921	905

Table 63. (Contd-2). PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/PROPANE MIXTURES

	<u>MCH</u>	<u>Propane</u>
Weight %	70	30
Mole %	51.2	48.8
Average Molecular Weight		71.8
Melting Point		-232°F
Heat of Formation (Liquid at 25°C)		-936 Btu/lb
Net Heat of Combustion (25°C)		18,990 Btu/lb
<u>Pseudocritical Properties</u>		
Pressure		550 psia
Temperature		392°F
Compressibility Factor		0.264
Acentric Factor		0.199

Temp., °F	Vapor Pressure, psia	Liquid Specific Gravity	Liquid Viscosity, cp	<u>Enthalpy, Btu/lb</u>	
				0 psia	1000 psia
-200		0.818	6.55	123	-69
-100	1.42	0.755	1.28	141	-39
0	18.4	0.707	0.494	167	0
100	91.6	0.641	0.281	202	49
200	289.	0.498	0.189	242	104
300				289	166
400				342	235
500				399	310
600				462	397
700				530	481
800				602	569
900				678	654
1000				758	739
1100				842	825
1200				929	914

Table 64. PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/n-BUTANE MIXTURE

	<u>MCH</u>	<u>n-Butane</u>
Weight %	90	10
Mole %	84.2	15.8
Average Molecular Weight		91.9
Melting Point		-207°F
Heat of Formation (Liquid at 25°C)		-859 Btu/lb
Net Heat of Combustion (25°C)		18,740 Btu/lb
<u>Pseudocritical Properties</u>		
Pressure		512 psia
Temperature		528°F
Compressibility Factor		0.255
Acentric Factor		0.237

Temp., °F	Vapor Pressure, psia	Liquid Specific Gravity	Liquid Viscosity, cp	<u>Enthalpy, Btu/lb</u>	
				<u>0 psia</u>	<u>1000 psia</u>
-200		0.879	29.2	123	-59
-100		0.830	3.52	139	-35
0	1.21	0.781	1.034	164	0
100	9.5	0.730	0.473	196	44
200	40.6	0.675	0.277	236	95
300	123.	0.583	0.192	281	153
400				333	219
500				389	293
600				451	380
700				518	463
800				589	552
900				664	636
1000				743	721
1100				826	807

Table 64 (Contd-1). PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/n-BUTANE MIXTURE

	<u>MCH</u>	<u>n-Butane</u>			
Weight %	80	20			
Mole %	70.3	29.7			
Average Molecular Weight		86.3			
Melting Point		-218°F			
Heat of Formation (Liquid at 25°C)		-886 Btu/lb			
Net Heat of Combustion (25°C)		18,820 Btu/lb			
<u>Pseudocritical Properties</u>					
Pressure		517 psia			
Temperature		492°F			
Compressibility Factor		0.258			
Acentric Factor		0.231			
Temp., °F	Vapor Pressure, psia	Liquid Specific Gravity	Liquid Viscosity, cp	<u>Enthalpy, Btu/lb</u>	
				<u>0 psia</u>	<u>1000 psia</u>
-200		0.860	18.1	122	-63
-100		0.810	2.54	139	-37
0	2.22	0.758	0.817	164	0
100	16.5	0.706	0.399	198	45
200	65.9	0.646	0.245	237	98
300	188.	0.528	0.175	284	158
400				336	224
500				393	299
600				455	386
700				522	470
800				594	558
900				670	643
1000				749	728
1100				831	813

Table 64 (Contd-2). PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/n-BUTANE MIXTURE

	<u>MCH</u>	<u>n-Butane</u>
Weight %	70	30
Mole %	58.0	42.0
Average Molecular Weight		81.4
Melting Point		-227°F
Heat of Formation (Liquid at 25°C)		-912 Btu/lb
Net Heat of Combustion (25°C)		18,910 Btu/lb
<u>Pseudocritical Properties</u>		
Pressure		523 psia
Temperature		459°F
Compressibility Factor		0.261
Acentric Factor		0.226

Temp., °F	Vapor Pressure, psia	Liquid Specific Gravity	Liquid Viscosity, cp	<u>Enthalpy, Btu/lb</u>	
				0 psia	1000 psia
-200		0.841	11.8	121	-67
-100		0.790	1.90	138	-39
0	3.11	0.737	0.664	165	0
100	22.8	0.683	0.343	199	47
200	88.3	0.620	0.221	239	100
300	246.	0.482	0.161	286	162
400				339	230
500				397	306
600				459	392
700				526	476
800				598	565
900				675	649
1000				754	734
1100				837	820

Table 65. LIQUID PROPERTIES OF JP-7 JET FUEL AT SATURATION

TEMP, F	VAPOR PRESS, PSIA	ENTHALPY OF VAR, BTU/LB	ENTHALPY, BTU/LB	ENTROPY, BTU/LB-R	SPEC HEAT AT CONST P, BTU/LB-R	DENSITY, LB/CU FT	VISCOSITY, LB/FT-HR	THERMAL COND, BTU/FT-HR-R
100	0.005	148.7	-54.7	-0.1287	0.5143	47.4	2.50	0.07756
200	0.147	138.8	0.0	0.0	0.5810	44.8	1.23	0.06899
300	1.59	127.7	61.83	0.1139	0.6646	42.1	0.734	0.06313
400	8.76	114.6	129.3	0.2163	0.7193	39.3	0.494	0.05496
500	31.3	98.8	200.9	0.3091	0.7916	36.0	0.354	0.04670
600	84.4	78.1	277.9	0.3963	0.9243	31.9	0.259	0.04210
700	196.	44.4	369.8	0.4875	1.409	25.5	0.175	0.03695

Gas Properties of JP-7 follows

### COMPRESSIBILITY FACTOR

TIME, F	PRESSURE, PSIA										
	0	100	200	300	400	500	600	700	800	900	1000
100	1.0000+00	8.2964-01	7.2330-01	4.9369-01	3.3074-01	3.4099-01	3.7269-01	4.9359-01	4.9359-01	4.7537-01	5.1249-01
200	1.0000+00	8.7730-01	8.0800-01	6.8706-01	5.5820-01	4.0437-01	4.7413-01	4.8554-01	5.0496-01	5.3059-01	5.8040-01
300	1.0000+00	9.1147-01	8.0800-01	7.8786-01	7.0847-01	6.4247-01	6.0598-01	5.9638-01	6.0169-01	6.1396-01	6.3078-01
400	1.0000+00	9.2704-01	8.6593-01	8.5645-01	8.0322-01	7.3929-01	7.2533-01	7.0690-01	7.0217-01	7.0690-01	7.1731-01
500	1.0000+00	9.5586-01	9.0761-01	9.5861-01	8.9322-01	8.3323-01	8.1993-01	8.0384-01	7.9555-01	7.9473-01	8.0022-01
600	1.0000+00	9.6949-01	9.3716-01	9.0413-01	8.7208-01	8.4383-01	8.1993-01	8.0384-01	7.9555-01	7.9473-01	8.0022-01
700	1.0000+00	9.7952-01	9.5847-01	9.3775-01	9.1840-01	9.0193-01	8.8910-01	8.8045-01	8.7580-01	8.7472-01	8.7715-01
800	1.0000+00	9.7952-01	9.5847-01	9.3775-01	9.1840-01	9.0193-01	8.8910-01	8.8045-01	8.7580-01	8.7472-01	8.7715-01
900	1.0000+00	9.7952-01	9.5847-01	9.3775-01	9.1840-01	9.0193-01	8.8910-01	8.8045-01	8.7580-01	8.7472-01	8.7715-01
1000	1.0000+00	9.7952-01	9.5847-01	9.3775-01	9.1840-01	9.0193-01	8.8910-01	8.8045-01	8.7580-01	8.7472-01	8.7715-01

COMPRESSIBILITY FACTOR

TIME	1400	1100	1200	1300	1400	1500	1600
8	51249-01	5.5033-01	5.8037-01	6.2634-01	6.6418-01	7.0193-01	7.3965-01
9	516040-01	5.9187-01	6.2562-01	6.5993-01	6.9322-01	7.2703-01	7.6047-01
1000	513078-01	6.5893-01	6.7553-01	7.0459-01	7.3504-01	7.6577-01	7.9608-01
1100	711731-01	7.3079-01	7.6536-01	7.9536-01	8.2606-01	8.1200-01	8.4029-01
1200	810022-01	8.1032-01	8.2340-01	8.3824-01	8.5433-01	8.7253-01	8.9325-01
1300	517715-01	8.8347-01	8.9360-01	9.0626-01	9.2168-01	9.3765-01	9.5490-01

1503/87-ALISND

TEMP., F	PRESSURE, PSIA									
	100	200	300	400	500	600	700	800	900	1,000
0										
100	0.0000									
200	0.0000									
300	0.0000									
400	0.0000									
500	0.0000									
600	0.0000									
700	0.0000									
800	0.0000									
900	0.0000									
1,000	0.0000									
1,100	0.0000									
1,200	0.0000									
1,300	0.0000									
1,400	0.0000									
1,500	0.0000									
1,600	0.0000									
1,700	0.0000									
1,800	0.0000									
1,900	0.0000									
2,000	0.0000									
2,100	0.0000									
2,200	0.0000									
2,300	0.0000									
2,400	0.0000									
2,500	0.0000									
2,600	0.0000									
2,700	0.0000									
2,800	0.0000									
2,900	0.0000									
3,000	0.0000									
3,100	0.0000									
3,200	0.0000									
3,300	0.0000									
3,400	0.0000									
3,500	0.0000									
3,600	0.0000									
3,700	0.0000									
3,800	0.0000									
3,900	0.0000									
4,000	0.0000									
4,100	0.0000									
4,200	0.0000									
4,300	0.0000									
4,400	0.0000									
4,500	0.0000									
4,600	0.0000									
4,700	0.0000									
4,800	0.0000									
4,900	0.0000									
5,000	0.0000									
5,100	0.0000									
5,200	0.0000									
5,300	0.0000									
5,400	0.0000									
5,500	0.0000									
5,600	0.0000									
5,700	0.0000									
5,800	0.0000									
5,900	0.0000									
6,000	0.0000									
6,100	0.0000									
6,200	0.0000									
6,300	0.0000									
6,400	0.0000									
6,500	0.0000									
6,600	0.0000									
6,700	0.0000									
6,800	0.0000									
6,900	0.0000									
7,000	0.0000									
7,100	0.0000									
7,200	0.0000									
7,300	0.0000									
7,400	0.0000									
7,500	0.0000									
7,600	0.0000									
7,700	0.0000									
7,800	0.0000									
7,900	0.0000									
8,000	0.0000									
8,100	0.0000									
8,200	0.0000									
8,300	0.0000									
8,400	0.0000									
8,500	0.0000									
8,600	0.0000									
8,700	0.0000									
8,800	0.0000									
8,900	0.0000									
9,000	0.0000									
9,100	0.0000									
9,200	0.0000									
9,300	0.0000									
9,400	0.0000									
9,500	0.0000									
9,600	0.0000									
9,700	0.0000									
9,800	0.0000									
9,900	0.0000									
10,000	0.0000									

1.503/8701B/CUFT

TEMP.	1000	1100	1200	1300	1400	1500	PRES. - DE. PSIA
100	2.5549+01	2.6172+01	2.6705+01	2.7177+01	2.7600+01	2.7981+01	1600
150	2.14647+01	2.2545+01	2.3290+01	2.3927+01	2.4499+01	2.5022+01	2.5324+01
180	1.7914+01	1.9095+01	2.0073+01	2.0848+01	2.1522+01	2.2134+01	2.2711+01
190	1.6793+01	1.7916+01	1.8966+01	1.9815+01	1.8815+01	1.9535+01	2.0136+01
195	1.6419+01	1.7491+01	1.8432+01	1.9432+01	1.6285+01	1.7085+01	1.7801+01
200	1.6085+01	1.7171+01	1.8237+01	1.9439+01	1.4238+01	1.4992+01	1.5705+01

## AFAPL-TR-70-71

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ENTHALPY, BTU/LB		PRESSURE, PSIA						
		500	400	300	200	100	0	
100	9.3335+01							1000
200	1.5860+02							900
300	1.6973+02							800
400	3.1375+02							700
500	3.6651+02							600
600	4.1663+02							500
700	4.4088+02							400
800	5.1386+02							300
900	5.9022+02							200
1000	6.6936+02							100
1100	7.5258+02							0
1200	8.3769+02							
1300	9.2560+02							
1400	10.1633+02							
1500	11.0992+02							
1600	12.0640+02							
1700	13.0580+02							
1800	14.0815+02							
1900	15.1349+02							
2000	16.2179+02							
2100	17.3305+02							
2200	18.4737+02							
2300	19.6478+02							
2400	20.8522+02							
2500	22.0873+02							
2600	23.3534+02							
2700	24.6509+02							
2800	25.9802+02							
2900	27.3419+02							
3000	28.7365+02							
3100	30.1645+02							
3200	31.6263+02							
3300	33.1223+02							
3400	34.6530+02							
3500	36.2189+02							
3600	37.8205+02							
3700	39.4584+02							
3800	41.1331+02							
3900	42.8451+02							
4000	44.5949+02							
4100	46.3831+02							
4200	48.2093+02							
4300	50.0742+02							
4400	51.9784+02							
4500	53.9228+02							
4600	55.9081+02							
4700	57.9350+02							
4800	59.9944+02							
4900	62.0871+02							
5000	64.2139+02							
5100	66.3756+02							
5200	68.5730+02							
5300	70.8069+02							
5400	73.0781+02							
5500	75.3874+02							
5600	77.7356+02							
5700	80.1235+02							
5800	82.5519+02							
5900	85.0199+02							
6000	87.5280+02							
6100	90.0764+02							
6200	92.6653+02							
6300	95.2949+02							
6400	97.9654+02							
6500	100.6770+02							
6600	103.4299+02							
6700	106.2243+02							
6800	109.0604+02							
6900	111.9384+02							
7000	114.8585+02							
7100	117.8209+02							
7200	120.8258+02							
7300	123.8734+02							
7400	126.9639+02							
7500	130.0976+02							
7600	133.2746+02							
7700	136.4951+02							
7800	139.7594+02							
7900	143.0678+02							
8000	146.4205+02							
8100	149.8178+02							
8200	153.2599+02							
8300	156.7470+02							
8400	160.2794+02							
8500	163.8573+02							
8600	167.4809+02							
8700	171.1505+02							
8800	174.8664+02							
8900	178.6188+02							
9000	182.4179+02							
9100	186.2639+02							
9200	190.1570+02							
9300	194.0975+02							
9400	198.0857+02							
9500	202.1218+02							
9600	206.2061+02							
9700	210.3388+02							
9800	214.5202+02							
9900	218.7505+02							
10000	223.0299+02							

ENTHALPY, BTU/LB	PRESSURE, PSIA				
	1400	1500	1600	1700	1800
1000	4.926+02	4.786+02	4.652+02	4.521+02	4.391+02
1100	5.371+02	5.231+02	5.097+02	4.964+02	4.831+02
1200	5.820+02	5.680+02	5.546+02	5.412+02	5.278+02
1300	6.269+02	6.129+02	5.995+02	5.861+02	5.727+02
1400	6.718+02	6.578+02	6.444+02	6.310+02	6.176+02
1500	7.167+02	7.027+02	6.893+02	6.759+02	6.625+02
1600	7.616+02	7.476+02	7.342+02	7.208+02	7.074+02
1700	8.065+02	7.925+02	7.791+02	7.657+02	7.523+02
1800	8.514+02	8.374+02	8.240+02	8.106+02	7.972+02
1900	8.963+02	8.823+02	8.689+02	8.555+02	8.421+02
2000	9.412+02	9.272+02	9.138+02	9.004+02	8.870+02
2100	9.861+02	9.721+02	9.587+02	9.453+02	9.319+02
2200	10.310+02	10.170+02	10.036+02	9.902+02	9.768+02
2300	10.759+02	10.619+02	10.485+02	10.351+02	10.217+02
2400	11.208+02	11.068+02	10.934+02	10.800+02	10.666+02
2500	11.657+02	11.517+02	11.383+02	11.249+02	11.115+02
2600	12.106+02	11.966+02	11.832+02	11.698+02	11.564+02
2700	12.555+02	12.415+02	12.281+02	12.147+02	12.013+02
2800	13.004+02	12.864+02	12.730+02	12.596+02	12.462+02
2900	13.453+02	13.313+02	13.179+02	13.045+02	12.911+02
3000	13.902+02	13.762+02	13.628+02	13.494+02	13.360+02
3100	14.351+02	14.211+02	14.077+02	13.943+02	13.809+02
3200	14.800+02	14.660+02	14.526+02	14.392+02	14.258+02
3300	15.249+02	15.109+02	14.975+02	14.841+02	14.707+02
3400	15.698+02	15.558+02	15.424+02	15.290+02	15.156+02
3500	16.147+02	16.007+02	15.873+02	15.739+02	15.605+02
3600	16.596+02	16.456+02	16.322+02	16.188+02	16.054+02
3700	17.045+02	16.905+02	16.771+02	16.637+02	16.503+02
3800	17.494+02	17.354+02	17.220+02	17.086+02	16.952+02
3900	17.943+02	17.803+02	17.669+02	17.535+02	17.401+02
4000	18.392+02	18.252+02	18.118+02	17.984+02	17.850+02
4100	18.841+02	18.701+02	18.567+02	18.433+02	18.299+02
4200	19.290+02	19.150+02	19.016+02	18.882+02	18.748+02
4300	19.739+02	19.599+02	19.465+02	19.331+02	19.197+02
4400	20.188+02	20.048+02	19.914+02	19.780+02	19.646+02
4500	20.637+02	20.497+02	20.363+02	20.229+02	20.095+02
4600	21.086+02	20.946+02	20.812+02	20.678+02	20.544+02
4700	21.535+02	21.395+02	21.261+02	21.127+02	20.993+02
4800	21.984+02	21.844+02	21.710+02	21.576+02	21.442+02
4900	22.433+02	22.293+02	22.159+02	22.025+02	21.891+02
5000	22.882+02	22.742+02	22.608+02	22.474+02	22.340+02
5100	23.331+02	23.191+02	23.057+02	22.923+02	22.789+02
5200	23.780+02	23.640+02	23.506+02	23.372+02	23.238+02
5300	24.229+02	24.089+02	23.955+02	23.821+02	23.687+02



# GAS PROPERTIES OF JP-7

## ENTROPY, BTU/LB-R

TEMP, F	100	200	300	400	500	600	700	800	900	1000
100	1.3578-01									
200	2.1039-01									
300	2.8218-01									
400	3.5140-01									
500	4.1821-01									
600	4.8275-01									
700	5.4514-01	5.3909-01								
800	6.0549-01	5.9539-01	5.8442-01	5.7013-01	5.6753-01	5.6677-01	5.6656-01	5.6659-01	5.6671-01	5.6690-01
900	6.6386-01	6.5076-01	6.5201-01	6.4543-01	6.3950-01	6.3671-01	6.3536-01	6.3469-01	6.3435-01	6.3420-01
1000	7.2040-01	7.1527-01	7.1212-01	7.0853-01	7.0483-01	7.0180-01	6.9978-01	6.9853-01	6.9775-01	6.9726-01
1100	7.7313-01	7.7111-01	7.6884-01	7.6443-01	7.6397-01	7.6172-01	7.5987-01	7.5848-01	7.5747-01	7.5676-01
1200	8.2413-01	8.2467-01	8.2311-01	8.2130-01	8.1949-01	8.1778-01	8.1626-01	8.1499-01	8.1397-01	8.1318-01
1300	8.7449-01	8.7673-01	8.7529-01	8.7384-01	8.7240-01	8.7103-01	8.6977-01	8.6863-01	8.6770-01	8.6691-01

## ENTROPY, BTU/LB-R

TEMP, F	1000	1100	1200	1300	1400	1500	1600
900	5.6690-01	5.6713-01	5.6737-01	5.6763-01	5.6788-01	5.6814-01	5.6840-01
1000	6.3420-01	6.3415-01	6.3417-01	6.3424-01	6.3433-01	6.3445-01	6.3458-01
1100	6.9726-01	6.9695-01	6.9677-01	6.9666-01	6.9661-01	6.9659-01	6.9651-01
1200	7.5676-01	7.5625-01	7.5580-01	7.5563-01	7.5544-01	7.5532-01	7.5521-01
1300	8.1318-01	8.1256-01	8.1208-01	8.1171-01	8.1143-01	8.1120-01	8.1103-01
1400	8.6691-01	8.6625-01	8.6571-01	8.6527-01	8.6491-01	8.6462-01	8.6437-01

## FREE ENERGY, BTU/LB

TEMP, F	100	200	300	400	500	600	700	800	900	1000
100	-2.0430+02									
200	-2.6654+02									
300	-3.4009+02									
400	-4.1570+02									
500	-5.0417+02									
600	-5.9417+02									
700	-6.9441+02	-5.9647+02	-8.0234+02	-8.0448+02	-8.0699+02	-8.0910+02	-8.1076+02	-8.1318+02	-8.1409+02	-8.1484+02
800	-7.9880+02	-8.0650+02	-9.1195+02	-9.1348+02	-9.1513+02	-9.1678+02	-9.1823+02	-9.1946+02	-9.2049+02	-9.2136+02
900	-9.0928+02	-9.1054+02	-1.0274+03	-1.0286+03	-1.0288+03	-1.0309+03	-1.0321+03	-1.0347+03	-1.0347+03	-1.0354+03
1000	-1.0254+03	-1.0264+03	-1.1478+03	-1.1486+03	-1.1503+03	-1.1511+03	-1.1520+03	-1.1534+03	-1.1540+03	-1.1546+03
1100	-1.1471+03	-1.1478+03	-1.2747+03	-1.2759+03	-1.2762+03	-1.2771+03	-1.2776+03	-1.2787+03	-1.2791+03	-1.2795+03
1200	-1.2742+03	-1.2747+03	-1.4069+03	-1.4077+03	-1.4081+03	-1.4085+03	-1.4089+03	-1.4092+03	-1.4095+03	-1.4099+03
1300	-1.4065+03	-1.4069+03	-1.5407+03	-1.5407+03	-1.5408+03	-1.5408+03	-1.5408+03	-1.5408+03	-1.5408+03	-1.5408+03

## FREE ENERGY, BTU/LB

TEMP, F	1000	1100	1200	1300	1400	1500	1600
900	-8.1484+02	-8.1547+02	-8.1601+02	-8.1645+02	-8.1682+02	-8.1713+02	-8.1739+02
1000	-9.2208+02	-9.2271+02	-9.2323+02	-9.2367+02	-9.2403+02	-9.2434+02	-9.2459+02
1100	-1.0354+03	-1.0359+03	-1.0364+03	-1.0368+03	-1.0372+03	-1.0374+03	-1.0377+03
1200	-1.1546+03	-1.1550+03	-1.1554+03	-1.1558+03	-1.1561+03	-1.1563+03	-1.1565+03
1300	-1.2795+03	-1.2799+03	-1.2802+03	-1.2804+03	-1.2806+03	-1.2808+03	-1.2810+03
1400	-1.4101+03	-1.4103+03	-1.4105+03	-1.4107+03	-1.4108+03	-1.4109+03	-1.4109+03

GAS PROPERTIES OF JP-7

SPECIFIC HEAT AT CONSTANT PRESSURE, BTU/LB-R

TEMP, °F	0	100	200	300	400	500	600	700	800	900	1000
100	6.2598-01										
200	6.8259-01										
300	5.3537-01										
400	5.6448-01										
500	6.3006-01										
600	6.7238-01										
700	7.1152-01										
800	7.4768-01	7.3593-01	8.0102-01	9.8360-01	9.9321-01	9.2515-01	9.0148-01	8.4922-01	8.2154-01	8.7519-01	8.7215-01
900	7.8102-01	7.9324-01	8.1252-01	8.4222-01	9.1887-01	9.4042-01	9.1964-01	9.0486-01	8.9358-01	8.8933-01	8.8482-01
1000	8.1172-01	8.2051-01	8.3262-01	8.5025-01	8.7529-01	9.0105-01	9.1186-01	9.1007-01	9.0521-01	9.0064-01	8.9690-01
1100	8.3996-01	8.4640-01	8.5459-01	8.6508-01	8.7807-01	8.9205-01	9.0326-01	9.0897-01	9.1021-01	9.0925-01	9.0734-01
1200	8.6590-01	8.7069-01	8.7646-01	8.8335-01	8.9127-01	8.9980-01	9.0727-01	9.1293-01	9.1621-01	9.1756-01	9.1769-01
1300	8.8972-01	8.9325-01	8.9737-01	9.0207-01	9.0727-01	9.1266-01	9.1775-01	9.2201-01	9.2511-01	9.2705-01	9.2803-01

SPECIFIC HEAT AT CONSTANT PRESSURE, BTU/LB-R

TEMP, °F	1000	1100	1200	1300	1400	1500	1600
1000	6.7219-01	6.7903-01	6.8586-01	6.9269-01	6.9952-01	7.0635-01	7.1318-01
900	6.6422-01	6.7106-01	6.7790-01	6.8473-01	6.9156-01	6.9839-01	7.0522-01
800	6.5635-01	6.6319-01	6.7002-01	6.7685-01	6.8368-01	6.9051-01	6.9734-01
700	6.4848-01	6.5532-01	6.6215-01	6.6898-01	6.7581-01	6.8264-01	6.8947-01
600	6.4061-01	6.4745-01	6.5428-01	6.6111-01	6.6794-01	6.7477-01	6.8160-01
500	6.3274-01	6.3958-01	6.4641-01	6.5324-01	6.6007-01	6.6690-01	6.7373-01
400	6.2487-01	6.3171-01	6.3854-01	6.4537-01	6.5220-01	6.5903-01	6.6586-01
300	6.1700-01	6.2384-01	6.3067-01	6.3750-01	6.4433-01	6.5116-01	6.5799-01
200	6.0913-01	6.1597-01	6.2280-01	6.2963-01	6.3646-01	6.4329-01	6.5012-01
100	6.0126-01	6.0810-01	6.1493-01	6.2176-01	6.2859-01	6.3542-01	6.4225-01

SPECIFIC HEAT AT CONSTANT VOLUME, BTU/LB-R

TEMP, °F	0	100	200	300	400	500	600	700	800	900	1000
100	4.1735-01										
200	4.7137-01										
300	5.2410-01										
400	5.7223-01										
500	6.1647-01										
600	6.6110-01										
700	7.0029-01	7.1432-01	7.2835-01	7.4238-01	7.5641-01	7.7044-01	7.8447-01	7.9850-01	8.1253-01	8.2656-01	8.4059-01
800	7.3845-01	7.6672-01	7.9509-01	8.2346-01	8.5183-01	8.8020-01	9.0857-01	9.3694-01	9.6531-01	9.9368-01	10.2205-01
900	7.7660-01	8.0505-01	8.3346-01	8.6187-01	8.9028-01	9.1869-01	9.4710-01	9.7551-01	10.0392-01	10.3233-01	10.6074-01
1000	8.1475-01	8.4320-01	8.7161-01	9.0002-01	9.2843-01	9.5684-01	9.8525-01	10.1366-01	10.4207-01	10.7048-01	10.9889-01
1100	8.5290-01	8.8131-01	9.0972-01	9.3813-01	9.6654-01	9.9495-01	10.2336-01	10.5177-01	10.8018-01	11.0859-01	11.3700-01
1200	8.9105-01	9.1946-01	9.4787-01	9.7628-01	10.0469-01	10.3310-01	10.6151-01	10.8992-01	11.1833-01	11.4674-01	11.7515-01
1300	9.2920-01	9.5761-01	9.8602-01	10.1443-01	10.4284-01	10.7125-01	10.9966-01	11.2807-01	11.5648-01	11.8489-01	12.1330-01

SPECIFIC HEAT AT CONSTANT VOLUME, BTU/LB-R

TEMP, °F	1000	1100	1200	1300	1400	1500	1600
1000	6.0126-01	6.0810-01	6.1493-01	6.2176-01	6.2859-01	6.3542-01	6.4225-01
900	5.9339-01	6.0023-01	6.0706-01	6.1389-01	6.2072-01	6.2755-01	6.3438-01
800	5.8552-01	5.9236-01	5.9919-01	6.0602-01	6.1285-01	6.1968-01	6.2651-01
700	5.7765-01	5.8449-01	5.9132-01	5.9815-01	6.0498-01	6.1181-01	6.1864-01
600	5.6978-01	5.7662-01	5.8345-01	5.9028-01	5.9711-01	6.0394-01	6.1077-01
500	5.6191-01	5.6875-01	5.7558-01	5.8241-01	5.8924-01	5.9607-01	6.0290-01
400	5.5404-01	5.6088-01	5.6771-01	5.7454-01	5.8137-01	5.8820-01	5.9503-01
300	5.4617-01	5.5301-01	5.5984-01	5.6667-01	5.7350-01	5.8033-01	5.8716-01
200	5.3830-01	5.4514-01	5.5197-01	5.5880-01	5.6563-01	5.7246-01	5.7929-01
100	5.3043-01	5.3727-01	5.4410-01	5.5093-01	5.5776-01	5.6459-01	5.7142-01

GAS PROPERTIES OF JP-7  
RATIO OF SPECIFIC HEATS

TEMP, °F	PRESSURE, PSIA					
	100	200	300	400	500	600
100	1.0271+00					
200	1.0230+00					
300	1.0218+00					
400	1.0198+00					
500	1.0181+00					
600	1.0176+00					
700	1.0160+00					
800	1.0152+00					
900	1.0140+00					
1000	1.0140+00					
1100	1.0135+00					
1200	1.0135+00					
1300	1.0131+00					
1400	1.0126+00					
1500	1.0126+00					
1600	1.0302+00	1.0002+00	1.0339+00	1.1779+00	1.1115+00	1.0879+00
1700	1.0247+00	1.0002+00	1.0622+00	1.1161+00	1.1273+00	1.1014+00
1800	1.0214+00	1.0002+00	1.0404+00	1.0602+00	1.0801+00	1.0853+00
1900	1.0192+00	1.0273+00	1.0204+00	1.0413+00	1.0523+00	1.0605+00
2000	1.0176+00	1.0233+00	1.0311+00	1.0322+00	1.0369+00	1.0449+00
2100	1.0159+00	1.0206+00	1.0259+00	1.0269+00	1.0314+00	1.0357+00
2200	1.0155+00	1.0187+00	1.0226+00			

RATIO OF SPECIFIC HEATS

TEMP, °F	PRESSURE, PSIA					
	100	200	300	400	500	600
100	1.0000+00					
200	1.0000+00					
300	1.0000+00					
400	1.0000+00					
500	1.0000+00					
600	1.0000+00					
700	1.0000+00					
800	1.0000+00					
900	1.0000+00					
1000	1.0000+00					
1100	1.0000+00					
1200	1.0000+00					
1300	1.0000+00					
1400	1.0000+00					
1500	1.0000+00					
1600	1.0456+00	1.0476+00	1.0438+00	1.0438+00	1.0423+00	1.0423+00
1700	1.0525+00	1.0500+00	1.0476+00	1.0463+00	1.0448+00	1.0427+00
1800	1.0549+00	1.0514+00	1.0487+00	1.0463+00	1.0441+00	1.0423+00
1900	1.0549+00	1.0519+00	1.0485+00	1.0461+00	1.0441+00	1.0423+00
2000	1.0522+00	1.0493+00	1.0469+00	1.0447+00	1.0428+00	1.0411+00
2100	1.0522+00	1.0493+00	1.0469+00	1.0447+00	1.0428+00	1.0411+00
2200	1.0474+00	1.0456+00	1.0438+00	1.0422+00	1.0408+00	1.0392+00
2300	1.0449+00	1.0411+00	1.0390+00	1.0379+00	1.0369+00	1.0369+00

SONIC VELOCITY, FT/SEC

TEMP, °F	PRESSURE, PSIA						1000
	100	200	300	400	500	600	
100	4.0184+02						
200	4.3558+02						
300	4.6588+02						
400	4.9622+02						
500	5.2391+02						
600	5.5022+02						
700	5.7532+02						
800	5.9935+02						
900	6.2252+02						
1000	6.4463+02						
1100	6.6439+02						
1200	6.8126+02						
1300	7.0753+02						
1400	7.4955+02	4.2562+02	2.7040+02	3.6052+02	4.7805+02	5.6473+02	6.8703+02
1500	5.2552+02	5.0195+02	4.2517+02	3.6596+02	4.0701+02	4.9023+02	6.2057+02
1600	5.5797+02	5.5993+02	5.1195+02	4.7099+02	4.5947+02	5.4370+02	5.9931+02
1700	6.0518+02	6.0745+02	5.7673+02	5.5084+02	5.3778+02	5.4509+02	6.1169+02
1800	6.1806+02	6.4711+02	6.2807+02	6.1344+02	6.0592+02	6.2513+02	6.5236+02
1900	6.5744+02	6.8126+02	7.019+02	6.6278+02	6.6103+02	6.7977+02	7.3243+02
2000	6.9417+02						
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# GAS PROPERTIES OF JP-7

## JOULE-THOMSON COEFFICIENT, R/PSI

TEMP, F	0	100	200	300	400	500	600	700	800	900	1000
100	3.9528-01										
200	2.7084-01										
300	1.9617-01										
400	1.4818-01										
500	1.1575-01										
600	9.2706-02										
700	7.5783-02	1.4600-01	1.3827-01	2.5602-01	8.6671-02	3.7455-02	2.2074-02	1.4499-02	9.9800-03	6.9738-03	4.8273-03
800	6.3000-02	1.0235-01	8.9490-02	1.1071-01	1.2259-01	7.7247-02	4.2497-02	2.6316-02	1.7793-02	1.2476-02	8.9972-03
900	5.3175-02	7.5869-02	6.9490-02	7.1635-02	7.6216-02	6.9631-02	5.1970-02	3.5693-02	2.4782-02	1.7739-02	1.3022-02
1000	4.5451-02	5.7863-02	6.4408-02	5.2109-02	5.3492-02	5.1371-02	4.4774-02	3.5792-02	2.7374-02	2.0732-02	1.5763-02
1100	3.9271-02	4.6045-02	4.9215-02	4.0404-02	4.0613-02	3.9240-02	3.5947-02	3.1151-02	2.5844-02	2.0895-02	1.6703-02
1200	3.4249-02	3.7875-02	3.9250-02	4.0404-02	3.2861-02	3.2594-02	3.1463-02	2.9357-02	2.6414-02	2.2998-02	1.9526-02
1300	3.0114-02	3.1761-02	3.2502-02	3.2861-02							1.6303-02

## JOULE-THOMSON COEFFICIENT, R/PSI

TEMP, F	1000	1100	1200	1300	1400	1500	1600
400	4.8273-03	3.2175-03	1.9655-03	9.6406-04	1.4507-04	-5.3702-04	-1.1137-03
500	4.9973-03	6.5280-03	4.6899-03	3.2715-03	2.1454-03	1.2311-03	4.7471-04
600	1.3022-02	9.7106-03	7.2854-03	5.4457-03	4.0093-03	2.8805-03	1.9233-03
700	1.5783-02	1.2114-02	9.3542-03	7.2345-03	5.5716-03	4.2414-03	3.1584-03
800	1.8703-02	1.3307-02	1.0590-02	8.4355-03	6.6967-03	5.2830-03	4.1208-03
900	1.6303-02	1.3476-02	1.1073-02	9.0613-03	7.3842-03	5.9839-03	4.8090-03

## VISCOSITY, LB/FT-HR

TEMP, F	0	100	200	300	400	500	600	700	800	900	1000
100	1.2218-02										
200	1.4259-02										
300	1.6282-02										
400	1.8286-02										
500	2.0282-02										
600	2.2265-02										
700	2.4232-02	2.5819-02	2.9310-02	3.7760-02	6.0295-02	7.4502-02	8.4061-02	9.2767-02	1.0013-01	1.0601-01	1.1097-01
800	2.6192-02	2.7556-02	3.1122-02	3.4544-02	4.1321-02	5.1199-02	6.0465-02	6.8802-02	7.6543-02	8.3324-02	8.9150-02
900	2.8142-02	2.9346-02	3.2618-02	3.4793-02	3.8471-02	4.3491-02	4.9400-02	5.5400-02	6.1238-02	6.6981-02	7.2552-02
1000	3.0083-02	3.1169-02	3.4205-02	3.5838-02	3.8342-02	4.1511-02	4.5320-02	4.9458-02	5.3714-02	5.7964-02	6.2193-02
1100	3.2018-02	3.3007-02	3.5942-02	3.7269-02	3.9076-02	4.1419-02	4.4067-02	4.7036-02	5.0185-02	5.3436-02	5.6711-02
1200	3.3942-02	3.4856-02	3.7695-02	3.8837-02	4.1211-02	4.2087-02	4.4147-02	4.6373-02	4.8749-02	5.1253-02	5.3852-02
1300	3.5861-02	3.6711-02									

## VISCOSITY, LB/FT-HR

TEMP, F	1000	1100	1200	1300	1400	1500	1600
400	4.1107-01	1.1526-01	1.1917-01	1.2278-01	1.2617-01	1.2936-01	1.3235-01
500	5.9315-02	9.4368-02	9.6948-02	1.0266-01	1.0624-01	1.0948-01	1.1274-01
600	7.2558-02	7.7800-02	8.2673-02	8.6703-02	9.0391-02	9.3896-02	9.7343-02
700	8.2190-02	8.6426-02	7.0643-02	7.4703-02	7.8538-02	8.1941-02	8.4928-02
800	9.6711-02	5.9374-02	6.3227-02	6.6488-02	6.9754-02	7.2930-02	7.5930-02
900	5.3853-02	5.6495-02	5.9126-02	6.1731-02	6.4319-02	6.6903-02	6.9480-02

GAS PROPERTIES OF JP-7

THERMAL CONDUCTIVITY: BTU/FT-HR-R

TEMP, F	100	200	300	400	500	600	700	800	900	1000
100	8.4701-03									
200	8.5803-03									
300	1.0805-02									
400	1.3237-02									
500	1.5813-02									
600	1.8510-02									
700	2.1310-02	2.1831-02								
800	2.4193-02	2.4681-02	2.5522-02	3.1320-02	3.4023-02	3.5525-02	3.6140-02	3.6990-02	3.9105-02	3.9890-02
900	2.7143-02	2.7542-02	2.8037-02	3.0242-02	3.2339-02	3.4139-02	3.5699-02	3.6924-02	3.8112-02	3.9046-02
1000	3.0148-02	3.0508-02	3.0947-02	3.1506-02	3.3297-02	3.4574-02	3.5795-02	3.6924-02	3.7925-02	3.8977-02
1100	3.3195-02	3.3523-02	3.3903-02	3.4350-02	3.4861-02	3.5501-02	3.6112-02	3.6705-02	3.7285-02	3.7853-02
1200	3.6268-02	3.6573-02	3.7291-02	3.7714-02	3.8188-02	3.8691-02	3.9247-02	3.9840-02	4.0372-02	4.0905-02
1300	3.9365-02	3.9819-02	3.9957-02	4.0200-02	4.0545-02	4.1033-02	4.1550-02	4.2097-02	4.2552-02	4.3112-02

THERMAL CONDUCTIVITY: BTU/FT-HR-F

TEMP, F	1000	1100	1200	1300	1400	1500	1600
800	3.9889-02	4.0568-02	4.1157-02	4.1689-02	4.2176-02	4.2663-02	4.3031-02
900	3.9046-02	3.9864-02	4.0568-02	4.1188-02	4.1749-02	4.2302-02	4.2821-02
1000	3.8977-02	3.9895-02	4.0692-02	4.1349-02	4.1939-02	4.2459-02	4.2922-02
1100	3.9783-02	4.0576-02	4.1342-02	4.2059-02	4.2719-02	4.3271-02	4.3790-02
1200	4.1405-02	4.2057-02	4.2688-02	4.3305-02	4.3909-02	4.4452-02	4.4933-02
1300	4.3512-02	4.4065-02	4.4602-02	4.5122-02	4.5626-02	4.6119-02	4.6602-02

PRANDTL NUMBER

TEMP, F	100	200	300	400	500	600	700	800	900	1000
100	8.0438-01									
200	8.0575-01									
300	8.0578-01									
400	8.0737-01									
500	8.0819-01									
600	8.0869-01									
700	8.0911-01	8.7053-01								
800	8.0945-01	8.5570-01	1.3793+00	1.9038+00	2.0238+00	2.1289+00	2.2317+00	2.3182+00	2.3752+00	2.4234+00
900	8.0974-01	8.4525-01	1.0169+00	1.2555+00	1.4889+00	1.6286+00	1.7463+00	1.8502+00	1.9403+00	2.0203+00
1000	8.0998-01	8.3820-01	9.3893+01	1.0445+00	1.1769+00	1.3029+00	1.4083+00	1.5013+00	1.5881+00	1.6696+00
1100	8.1019-01	8.3339-01	9.0256+01	9.6521+01	1.0338+00	1.1271+00	1.2078+00	1.2828+00	1.3526+00	1.4187+00
1200	8.1037-01	8.2980-01	8.8283+01	9.2345+01	9.7587+01	1.0333+00	1.0913+00	1.1483+00	1.2030+00	1.2561+00
1300	8.1053-01	8.2707+01	8.6954+01	8.9750+01	9.3611+01	9.7775+01	1.0215+00	1.0637+00	1.1062+00	1.1526+00

PRANDTL NUMBER

TEMP, F	1000	1100	1200	1300	1400	1500	1600
800	2.4358-00	2.4694+00	2.5087+00	2.5454+00	2.5799+00	2.6124+00	2.6430+00
900	2.0203+00	2.0671+00	2.1432+00	2.1843+00	2.2228+00	2.2582+00	2.2935+00
1000	1.6896+00	1.7034+00	1.8111+00	1.8649+00	1.9131+00	1.9583+00	2.0021+00
1100	1.4187+00	1.4823+00	1.5447+00	1.6029+00	1.6564+00	1.7031+00	1.7431+00
1200	1.2569+00	1.3079+00	1.3573+00	1.4056+00	1.4529+00	1.4980+00	1.5399+00
1300	1.1486+00	1.1932+00	1.2305+00	1.2694+00	1.3073+00	1.3445+00	1.3810+00

# GAS PROPERTIES OF JP-7 AT SATURATION

TEMP, F	VAPOR PRESS, PSIA	COMP FACTOR	DENSITY, LB/CUFT	FUGACITY, PSIA	ENTHALPY, BTU/LB	ENTROPY, BTU/LB-R	FREE ENERGY, BTU/LB	SPEC HEAT AT CONST P, BTU/LB-R
100	4.9871-03	9.9995-01	1.4638-04	4.9238-03	9.3332+01	1.3578-01	-2.0430+02	4.2599-01
200	1.4862-01	9.9996-01	3.7216-03	1.4834-01	1.3874+02	2.1031-01	-2.6854+02	4.2264-01
300	1.5746+00	9.9223-01	3.4455-02	1.5625+00	1.8929+02	2.8170-01	-3.4016+02	5.3706-01
400	8.6943+00	9.6818-01	1.7238-01	8.4292+00	2.4408+02	3.4961-01	-4.1909+02	5.3075-01
500	3.1137-01	9.1082-01	5.9111-01	2.8805+01	3.0206+02	4.1455-01	-5.0504+02	6.4480-01
600	8.5279+01	8.0231-01	1.6544+00	7.8012+01	3.6158+02	4.7530-01	-5.9826+02	7.0505-01
700	1.5008+02	6.0200-01	4.5987+00	1.3738+02	4.1845+02	5.2966-01	-6.9868+02	8.2012-01

# GAS PROPERTIES OF JP-7 AT SATURATION

TEMP, F	VAPOR PRESS, PSIA	SPEC HEAT AT CONST V, BTU/LB-R	RATIO OF SPEC HEATS	SOUND VEL, FT/SEC	J-T COEF, R/PSI	VISCOSITY, LB/FT-HR	THERMAL COND, BTU/FT-HR-R	PRANDTL NUMBER
100	4.9871-03	4.1476-01	1.0271+00	4.0183+02	1.4470+00	1.2218-02	6.4701-03	8.0455-01
200	1.4862-01	4.7157-01	1.0239+00	4.3514+02	8.4869-01	1.8262-02	8.5414-03	8.0624-01
300	1.5726+00	5.2535-01	1.0219+00	4.6335+02	5.1346-01	1.6311-02	1.0815-02	8.1001-01
400	8.6943+00	5.7831-01	1.0215+00	4.6070+02	3.3300-01	1.8436-02	1.3287-02	8.1968-01
500	3.1403+01	6.2961-01	1.0241+00	4.7709+02	2.3790-01	2.0799-02	1.5988-02	8.3682-01
600	8.5278+01	6.8163-01	1.0344+00	4.3708+02	2.0653-01	2.3789-02	1.9009-02	8.8234-01
700	1.9081+02	7.3015-01	1.0453+00	3.1849+02	2.6652-01	2.9283-02	2.2739-02	1.0561+00

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<b>13. ABSTRACT</b> The general objective of this investigation is the development of new fuels and new fuel systems which will provide the cooling and propulsion requirements of advanced air-breathing engines. In previous studies on the utilization of endothermic reactions of hydrocarbon fuels, the catalytic dehydrogenation of naphthenes showed the most promise for practical applications. In continuing these studies, a large number of catalysts were prepared and tested for activity and stability for the dehydrogenation of methylcyclohexane and decalin. The stability of supported platinum catalysts for these reactions was affected by the physical properties and chemical composition of the support and by the metal content and metal composition of the catalyst. Bicyclo(2,2,2)octane was dehydrogenated to bicyclooctene in low yield with a supported platinum catalyst. Additives were found which enhanced the rate of thermal cracking of paraffins. Improved wall catalysts were formulated for the dehydrogenation of naphthenes and a mathematical model was developed for a catalytic wall reactor. Various fuels were evaluated for thermal stability in both our standard ASTM Coker and a special Alcor JFTOT unit designed for operation up to 1000°F and 1000 psi. The high temperature thermal stability of fuels was improved by means of additives. The deposit profiles on coker tubes were reproducibly determined with a recently developed beta-ray backscatter instrument which is capable of measuring deposit thicknesses up to 2500 Å. The physical properties of JP-7 jet fuel were recalculated using improved methods. The supersonic combustion of decalin, tetralin and naphthalene was investigated using the shock tube. A literature survey was made of articles		

DD FORM 1473 and patents of interest to this and related programs.

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